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**SPACE PROCESSES FOR EXTENDED LOW-G TESTING**

**GENERAL DYNAMICS**  
*Convair Aerospace Division*

REPORT NO. CASD-STT-73-005

# **SPACE PROCESSES FOR EXTENDED LOW-G TESTING**

## **FINAL REPORT**

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National Aeronautics and Space Administration  
Process Engineering Laboratory  
GEORGE C. MARSHALL SPACE FLIGHT CENTER  
Huntsville, Alabama

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San Diego, California



## FOREWORD

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## SUMMARY

The objective of this study was to investigate the feasibility of verifying the capabilities of space processes in ground-based experiments at extended low-g periods. This was accomplished by a detailed evaluation of 18 typical processes with regard to the effectiveness of limited-time experiments and the required facilities. It was found that for 17 of the 18 evaluated processes, a valid representation of the complete process cycle can be achieved at low-g periods ranging from 40 to 390 seconds, typical for land-based trajectories of commonly used research (sounding) rockets. For a limited number of processes, specific process parameters may be verified in drop tower or aircraft experiments with low-g time capabilities from 3 to 8 seconds.

In the course of the process and experiment studies, a minimum equipment inventory was defined, consisting of a limited number of multi-purpose processing devices and a generally applicable support module. A modular equipment design was adopted which assures low cost and a high degree of program flexibility.

Procedures and data were established for the synthesis and definition of dedicated and mixed rocket payloads, accommodating an average of 4 to 5 experiments in each flight. A typical plan for the initial phase of a continuing rocket test program was formulated, consisting of 10 flights obtainable with 5 rockets and appropriate refurbishment. It extends over a period of two years, including equipment development, a 12-month period of launch operations and the evaluation of results. The 10-flight program covers 17 of the 18 candidate processes and comprises 45 experiments (processing conditions), producing a total of 64 samples (material compositions) for evaluation.

It is expected that the results of such programs provide valuable data and experience for the definition of shuttle-based experiments and facilities.

## 1. STUDY OBJECTIVE AND SCOPE OF THIS REPORT

The objective of this study was to define the space processes and individual process parameters that can be evaluated and verified in low-g test facilities with short and extended low-g time capabilities, to specify individual experiments and to formulate a two-year experiment program.

The study was carried out in three consecutive tasks as follows:

- Task I: Selection of processes and materials for detailed study.
- Task II: For the processes selected in Task I, definition of low-g experiments, test facilities and experiment protocols.
- Task III: Development and specification of experiment plans and formulation of a two-year experiment program.

A preliminary evaluation of all known low-g processes was documented in an interim report of Aug. 2, 1972. A summary of this evaluation and the identification of the processes selected for this study are presented in Section 2, Technical Approach. This section further contains a discussion of the approach to Tasks II and III.

Task II studies and results are documented in Sections 3 through 7. The first four of these sections comprise topics of a generally applicable nature: Adopted Basic Concepts (Sect. 3), Low-g Test Facilities (Sect. 4), Experiment Payload Packages (Sect. 5) and Heating and Cooling Methods and Devices (Sect. 6). This is followed by the detailed definition of experiments for each of the 18 selected processes (Sections 7.1 through 7.18). For convenient reference, a standard format is used for all process evaluations. The 9-subject format is detailed in the Technical Approach, pages 2-4/2-5.

Task III studies and results are documented in Sections 8 and 9. Section 8 establishes the procedures and data for the definition of dedicated and mixed payloads.

The sequencing of such payloads into effective programs are discussed in the initial part of Section 9. A plan for a typical 2-year program, representing the initial phase of a continuing rocket test program is presented in Sections 9.4 and 9.5.

The results of the study as to the effectiveness of extended low-g experiments for the verification of process capabilities are summarized in the Conclusions, Section 10.

## 2. TECHNICAL APPROACH

### 2.1 TASK I: SELECTION OF PROCESSES

A preliminary evaluation of all known processes was carried out earlier in the program whose results were documented in an interim report (Progress Report #2, Aug. 20, 1972). It consisted of a discussion of each process, the definition of its significant verification requirements and a preliminary classification of processes with regard to adaptability to low-g testing. The results of the evaluation were summarized in several charts which are included in this report (Tables 2 and 3). On the basis of this evaluation, the processes listed in Table 1 were selected for detailed study and the definition of low-g test requirements.

In Table 1 the processes are arranged in accordance with the classification system established by MSFC. The first-order classification is by material category as follows:

Category I	Biological Materials
II	Composite Materials
III	Alloys and Immiscible Systems
IV	Glasses
V	Single Crystal Materials
VI	General Materials R&T

Only one process was selected for Category V - Single Crystal Materials because it is not readily adaptable to low-g experiments within the two-year time-frame; however, the selected process (zone melting) provides all essential data on the characteristics of zero-g grown single crystals representative of other processes of this category. The rationale for these exclusions/limitations is discussed in the first interim report and identified in summary form in Table 2.

The second-order classification is by the primary g-sensitive processing phase, identifying the following nine "Process Groups."

1. Contact-free and Captive Suspension Processes
2. Mixing and Homogenization Processes
3. Separation and Purification Processes
4. Heating and Melting Processes
5. Cooling and Solidification Processes
6. Shaping and Forming Processes
7. Single Crystal Processes
8. Chemical Processes
9. Biological Processes.

Frequently the prime objective of the process is identified by the process group, rather than the material (product) category, as in the case of forming and shaping processes which apply to more than one material category; in the listing of Table 1 this process group is placed under Category VI - General R&T, since it is considered as a generally applicable processing method.

With the adoption of this classification, the code numbers which have been introduced in Task I for the identification of individual processes have no longer any classifying meaning. They are, however, retained - merely for the purpose of identification and convenient reference.

## 2.2 APPROACH TO TASK II: PROCESS EVALUATION AND EXPERIMENT DEFINITION

The process studies, documented in Section 7.0, consisted of 1) the definition of process verification requirements in terms of product characteristics to be verified, experimental materials, samples and the required low-g test time, 2) the definition of low-g test facilities and experiment apparatus and 3) the specification of experiment procedures and operational requirements. All definitions are in such depth as to



firmly establish the feasibility and effectiveness of process verification by extended-time low-g testing, to facilitate the formulation of a test program and to provide the data and rationale which substantiate such a program.

This was accomplished by an iterative approach, consisting of three major phases as illustrated in Fig. 2-1. First, all selected processes were analysed with regard to:

- (1) Clear identification of each process, the product characteristics of significance in applications, and the expected gains in these characteristics by processing in zero-g.
- (2) Verification objectives - Zero-g process characteristics and product properties to be verified in experiments. Measurement and representation of these characteristics and properties. If indicated, definition of several degrees of verification accuracy ("verification levels").
- (3) Evaluation of applicable materials and identification of most promising materials for experiments and the verification objectives (2).
- (4) Definition of minimum material quantities which permit an adequate measurement of the properties to be verified.
- (5) Evaluation of processes and conceptual definition of "experimental" processes and procedures which are adaptable to the limitations of extended-time low-g testing, yet at the same time yield adequate data as to processing parameters and product properties.
- (6) Evaluation of experimental processing methods. Selection of most effective methods and devices. Conceptual apparatus design studies.
- (7) Numerical assessment of the performance characteristics of the methods and devices defined in (6), such as thermodynamic data, power requirements, dimensional requirements, weight etc.
- (8) Establishment of data on the capabilities of low-g test facilities with emphasis on facilities for extended low-g time (rockets), such as low-g time, payload weight and payload dimensions.

The second phase of the Task II analysis had the objective to arrive at specific definitions and requirements as to materials, samples, processes and devices for

low-g experiments. It consisted of the following operations (numbering of items continued for positive reference):

- (9) Trade-off studies between specific materials, sample sizes and configurations, processing methods, processing devices, operational requirements and low-g time, using data established in (3) through (8) above. This included extensive thermodynamic computations and equipment design studies.
- (10) Adoption of basic concepts. The experiences and data accrued in the foregoing studies, particularly in (9) above, indicated the desirability of specific approaches to experiment planning, experiment definition and equipment design. A number of "basic concepts" were adopted as guidelines for the subsequent studies. In view of the basic importance - in the opinion of the contractor - of these concepts for space manufacturing experiments in general, a special section (3.0) is devoted to their discussion.
- (11) Final selection of specific experiment elements, such as material(s), sample configuration, processing procedure, low-g time requirement and apparatus components.
- (12) Preliminary payload assembly studies for the purpose of establishing guidelines for apparatus (modules) envelope limitations and weight distribution.

In the final third phase of Task II, detailed experiment specifications and apparatus designs were developed and defined for each process and verification level. Each specification consists of the following:

- (1) Definition of the basic process and its objectives. Definition of the experimental process(es) and the verification level(s).
- (2) Definition of verification requirements in terms of measurements.
- (3) Definition of experimental materials and significant materials data, such as processing temperature.
- (4) Definition of experimental material quantity and sample configuration.
- (5) Definition of the experimental process and process phases. Identification of g-sensitive phases.

- (6) Definition of low-g test requirements for the defined material (3), sample size (4), and low-g processing phases (5) comprising:
  - (a) Experiment time requirements.
  - (b) Operational requirements, such as heat and power requirements.
- (7) Definition of low-g experiments, consisting of:
  - (a) Correlation of the requirements defined in (6) with low-g facility capabilities.
  - (b) Selection of the most effective low-g test facility.
  - (c) By comparison of experiment apparatus requirements and the payload capability of the facility (b), definition of the number of samples which can be processed in one test (flight).
- (8) Definition of the experiment payload comprising:
  - (a) Apparatus (processing module) design.
  - (b) Apparatus assembly.
  - (c) Support equipment (support module).

Definitions include configurations, dimensions and weight.

- (9) Definition of experiment performance, comprising pre-flight, flight and post-flight operations.

In the course of the design studies, a number of attractive, yet unconventional concepts were conceived. They were, however, discarded for the time being in favor of state-of-art designs in order to assure unquestionable feasibility. The consideration of more sophisticated concepts should be left to individual experiment and hardware development programs as they will evolve from the activation of the low-g test plan.

## 2.3 PRESENTATION OF TASK II RESULTS

The results of the Task II studies are presented in Sections 3.0 through 7.0. The first four of these sections deal with the following basic and generally applicable subjects.

- 3.0 Adopted Basic Concepts
- 4.0 Capabilities of Low-g Test Facilities
- 5.0 Experiment Payload Packages
- 6.0 Heating and Cooling.

These subjects are discussed separately in order to preclude repetitious statements in the experiment definitions. Sections 3.0 through 5.0 apply to all experiments; Section 6.0 applies to 16 of the 18 defined experiments, excepting the biological separation of biochemicals. The subsequent section

### 7.0 Process Analysis and Experiment Definition

contains the evaluation of the 18 selected processes and the definition of experiment specifications. The sub-section numbers under which each process is discussed are identical with the process identification numbers in Table I.

## 2.4 APPROACH TO TASK III. EXPERIMENT PLANS AND PROGRAMS

In the individual process evaluations of Task II it was demonstrated that more than one - up to six - experiments can be accommodated in one payload or flight. The procedures developed in the definition of such "dedicated" payloads were generalized in Task III so that they can also be applied to the definition of payloads comprising two or more processes, designated as "mixed" payloads. They consist essentially in the trade-off of functional and physical experiment requirements, such as low-g time, power requirements and physical equipment characteristics against the corresponding capabilities of the vehicle and of the support module which matches the mixed equipment requirements. All data necessary for the synthesis of payloads were

extracted from the process evaluations of Task II and tabulated for convenient accessibility. They were arranged into four groups of "payload elements" as follows:

- (1) Experiment requirements (functional, processing equipment)
- (2) Characteristics of individual processing modules
- (3) Functional capabilities and physical requirement of support modules
- (4) Rocket capabilities

The definition of mixed payloads further called for the assessment of experiment compatibility, determined by the functional and dimensional compatibility of processing modules and by their adaptability to a common support module type.

The results of the payload definition studies and all tabular information required for the development of mixed payloads are presented in Section 8.

The first step toward the development of test programs was the definition of program effectiveness criteria and constraints. This was followed by the establishment of procedures for the most effective sequencing of payloads into a multi-flight program. One of the first-order criteria for payload sequencing is the availability of the concerned equipment. The earliest time of availability in months from program start was defined for each module of the equipment inventory evolving from Task II.

On the basis of these procedures and data, various choices for an initial rocket test program were formulated and evaluated for effectiveness in terms of the number of represented processes and the number of experiments (processing conditions) and samples (material compositions) for each process, all in relation to the required number of flights. The most effective choice which emerged from these evaluations was a 10-flight program, using 5 rockets with proper refurbishment.

The selected program was considered as a typical first phase of a continuing test program. It extends over a two-year period, including the time required for equipment development, flight operations and test evaluation. Detailed plans and schedules were formulated for this program and the related equipment development.

The program studies are presented in Section 9. All Task III studies were confined to rocket experiments; drop tower experiments were defined in Task II for the applicable processes.

Table 1

PROCESSES SELECTED FOR DETAILED STUDY

CATEGORY I: BIOLOGICAL MATERIALS

- 1 Electrophoretic Separation of Biochemicals - Stationary
2. Electrophoretic Separation of Biochemicals - Continuous (EMP)

CATEGORY II: COMPOSITE MATERIALS

- 3 Fiber/Particle Composites - Predispersed
- 4 Fiber/Particle Composites - Low-g Mixing
- 5 Controlled Density Metals - Predispersed
- 6 Controlled Density Metals - Dynamic Foaming
- 7 Unidirectional Eutectics

CATEGORY III: ALLOYS AND IMMISCIBLE SYSTEMS

- 8 Superconductors - Predispersed
- 9 Metastable Alloys - Thermal Dispersion - Moderate Temp.
- 10 Metastable Alloys - Thermal Dispersion - High Temperatures
- 11 Metastable Alloys - Low-g Homogenization

CATEGORY IV: GLASSES

- 17 Oxide Glasses
- 18 Chalcogenide Glasses

CATEGORY V: SINGLE CRYSTAL MATERIALS

- 12 Single Crystal Growth - Zone Melting
- 13 Kinetics of Nucleation and Crystal Growth

CATEGORY VI: GENERAL MATERIALS R & T

- 14 Containerless Alloying
- 15 Free Processing System
- 16 Drawing of Membranes

Table 2. Basic Characteristics of Candidate Processes -- Composites

Table 1: Comprehensive Data for Material Analysis																				
1		2			3		4			5				6		7				
Ident. Code	Classification *	Product/Process			Primary Low-G Effects	Verif		G-Sensitive Process Elements	Verif		Fund Subj	Non-G-Sensitive Process Elements	Fund Subj	Cap Gain LG/G Appl	Prod Assu Succ	Grow Pot	Prod Val	Total		
		A	C	B <sub>1</sub>		B <sub>2</sub>	G		LG	AN									G	LG
A1-1	1.2 25 01 32 34 42	1.2	25	01	Metal-Matrix Fiber/Particle Composites (Predispersed Compact)	Mixture Stability	o	•	Mixing of Solids Melt Cycle Casting of Mixture	o	•	H1	Reinf. Surface Treatm. Compact Preparation	H5	2	3	2	2	2.5	
A1-2	1.2 25 01 32 34 42	1.2	25	01	Metal-Matrix Fiber/Particle Composites (Dynamic Dispersion)	Mixture Stability Uniform Dispersion	o	•	Mixing Dispersion Control Casting of Mixture	o	•	H2, G3 H2, G3 H6	Reinf. Surface Treatm. Reinf. Immersion		2	3	2	3	2.5	
A2-1	1.4 21 01 32 34 42	1.4	21	01	Controlled Density Metals (Predispersed Compact Thermal Gas Evolution)	Mixture Stability	•	•	Mixing of Solids Melt Cycle Gas Evolution Control	o	•	H1	Reinf. Surface Treatm. Compact Preparation	H5	3	3	3	3	3	
A2-2	1.4 21 01 32 34 42	1.4	21	01	Controlled Density Metals (Dynamic Foaming)	Mixture Stability Uniform Dispersion of Gas and Reinforc.	•	•	Foaming/Mixing Bubble Size and Dispersion Control	o	•	H2, G3	Reinf. Surface Treatm. Bubble Surface Stabilization Reinf. /Gas Mixing	G3, H2	3	3	2	3	3	
A2-3	1.4 21 01 32 34 42	1.4	21	01	Controlled Density Metals (Ultrasonic Foaming)	Mixture Stability Uniform Gas Dispersion	•	•	Foaming/Mixing Bubble Size and Dispersion Control	o	•	H2, G3	Dissolution of Gases Ultrasonic Techniques		2	2	1	2	2	
A2-4	1.4 21 01 32 34 42	1.4	21	01	Controlled Density Metals (Nucleate Foaming)	Mixture Stability Uniform Gas Dispersion	•	•	Dispersion of Nuclei Bubble Size Control	o	•	H2	Preparation of Nuclei Melt Pressure Control		2	2	1	2	2	
A3	1.1 40 00 32 34 42	1.1	40	00	Unidirectional Eutectics	Zero-Convection Zero-Segregation	•	•	Unidirectional Solidification of Eutectic Phase	•	•	G1	Therm. Gradient Control Progression Rate Contr.		3	2	3	2	2.5	
A4-1	1.0 55 01 32 34 42	1.0	55	01	Supersaturated Alloys (Thermal Dispersion)	Stability of Dispersion	•	•	Thermal Generation of Dispersed Phase	o	•	G2	Alloy Preparation Thermal Control		3	2	3	2	2.5	
A4-2	1.0 55 01 32 34 42	1.0	55	01	Supersaturated Alloys (Dynamic Dispersion)	Stability of Dispersion	•	•	Homogenization	o	•	H2, G3	Preparation of Pre-Mix Cooling Rate Control	H1, H2	3	2	3	3	3	

\* M. S. F. C. CLASSIFICATION SYSTEM

o CONCLUSIVE VERIFICATION  
o DEMONSTRATION OR PARTIAL VERIFICATION

1 = LOWEST  
3 = HIGHEST



Table 2. Basic Characteristics of Candidate Processes – Alloys and Single Crystals

1		2		3		4		5				6		7							
Ident. Code	Classification *				Product/Process		Primary Low-G Effects	Verif G LG	G-Sensitive Process Elements		Verif		Fund Subj	Non-G-Sensitive Process Elements	Fund Subj	Process Potential					
	A	C	B <sub>1</sub>	B <sub>2</sub>							AN	G				LG	Cap LG/G	Prod Appl	Assu Succ	Grow Pot	Prod Val
B1-1	1.0	01	03	32	Contact-Free Alloying		Free Suspension Containment by Surface Tension	•	Position Control Removal of Gases		•	•	H4	Compact Preparation	H5	3	2	3	1	2	2.5
B2-1	3.2	30	01	31	Superconducting alloys (Predispersed Compact)		Zero-Segregation Zero-Convection	•	Mixing of Solids Removal of Gases		•	•	H1	Compact Preparation	H5	3	3	2	2	3	2.5
B2-2	3.2	30	03	32	Superconducting Alloys (Induction Mixing)		Zero-Segregation Zero-Convection Contact-Free Suspension	•	Induction Mixing Removal of Gases		•	•	H2, G3	Preparation of Ingots (Segregated)		3	3	2	3	3	3
B3	1.1	50	01	34	Thermosetting Alloys		Mixture Stability	•	Mixing		•		H2	Compression Solubility		2	3	1	2	1	1.5
C1-1	3.1	31	01	34	Single Crystal Growth From Solution (Aqueous Solutions)		Zero-Convection Mixture Stability	•	Mixing Dopant Dispersion Contr.		•	•	H2	Temp. Gradient Control		2	2	3	1	2	2
C1-2	3.1	31	01	34	Single Crystal Growth From solution (Molten Solutions)		Zero-Convection Mixture Stability	•	Mixing Dopant Dispersion Contr.		•	•	H2	Prep. of Pre-mixed Solution Material Temp. Gradient Control		2	3	3	2	2	2.5
C2-1	3.1	32	05	32	Single Crystal Growth From the Melt (Zone Melting)		Zero-Convection	•	Shape Control		•	•	G2	Temp. Gradient Control Progression Rate Control		3	3	2	2	3	2.5
C2-2	3.1	33	05	31	Single Crystal Growth From the Melt (Crystal Pulling)		Zero-Convection	•	Surface Tension-Shape Control		•	•	G2	Temp. Gradient Contr. Shape/Progression/Current Control		3	3	2	3	3	3
C2-3	3.1	32	05	33	Single Crystal Wires or Ribbons (Continuous Zone Melting)		Zero-Convection	•	Surface Tension-Shape Control		•	•	G2	Temp. Gradient Contr. Progression Rate Contr.		2	3	2	2	3	2.5
C3	3.1	34	01	34	Single Crystal Growth From the Vapor		Zero-Convection	•	Solid/Gas Interface Effects				G2	Thermal Gradient Contr.		3	3	2	3	3	3

\* M. S. F. C. CLASSIFICATION SYSTEM

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o DEMONSTRATION OR PARTIAL VERIFICATION

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3 = HIGHEST

Table 2. Basic Characteristics of Candidate Processes – Glasses, Biochemicals and Isotopes

1	2				3	4			5			6		7							
Ident. Code	Classification *				Product/Process	Primary Low-G Effects	Verif		G-Sensitive Process Elements	Verif		Fund Subj	Non-G-Sensitive Process Elements	Fund Subj	Process Potential						
	A	C	B <sub>1</sub>	B <sub>2</sub>			G	LG		AN	G				LG	Cap Gain LG/G	Prod Appl	Succ	Assu Grow	Pot	Prod Val
D1-1	2.0	45	03	32	Amorphous Oxides (Glasses)	Contact-Free Processing Containment by Surface Tension	•	•	Position Control	•	•	H4 G1	Contact-Free Heating and Cooling Dissolution of Nuclei	H4	3	3	3	3	3	3	3
D1-2	2.0	04	03	32	Shaping of Amorphous Oxides	Contact-Free Processing Shape Definition by Surface Tension	•	•	Position Control Induced Deformation	•	•	H4 G2	Contact-Free Heating and Cooling Force Field Control	H4 H4	2	3	1	1	1	3	2
E1-1	4.2	65	28	01	Electrophoretic Separation of Biochemicals - Stationary Column	Zero-Sedimentation Zero-Convection	•	•	Gas Removal at Elec- trodes	•	•	H3	Temperature Control Sample Deployment and Recovery		2	3	3	2	3	2.5	
E1-2	4.2	65	28	01	Electrophoretic Separation of Biochemicals - Continuous (Curtain Type)	Zero-Sedimentation Zero-Convection	•	•	Buffer Flow Contr. Gas Removal at Electrodes	•	•	H3	Temperature Control Continuous Sample Feeding and Recovery		2	3	2	1	3	2	
E1-3	4.2	65	28	01	Separation of Biochemicals by Continuous Electro- Magnetophoresis	Zero-Sedimentation Zero-Convection	•	•	Buffer Flow Control Gas Removal at Electrodes	•	•		Temperature Control Continuous Sample Feeding and Recovery		3	3	3	3	3	3	
E2-1	5.0	60	28	01	Electrophoretic Separation of Isotopes	Zero-Sedimentation Zero-Convection	•	•	Flow Control	•	•				1	2	2	2	3	2	
E2-2	5.0		21	01	Centrifugal Separation of Isotopes	Zero-Convection Weightlessness	•	•							1	3	1	1	3	1.5	

\* M. S. F. C. CLASSIFICATION SYSTEM

• CONCLUSIVE VERIFICATION  
o DEMONSTRATION OR PARTIAL  
VERIFICATION

1 = LOWEST  
3 = HIGHEST



Table 2. Basic Characteristics of Candidate Processes – Fundamental and Process Research

1	2		3		4		5				6		7							
Ident. Code	Classification *				Product/Process	Primary Low-G Effects	Verif		G-Sensitive Process Elements	Verif		Fund Subj	Non-G-Sensitive Process Elements	Fund Subj	Process Potential					
	A	C	B <sub>1</sub>	B <sub>2</sub>			G	LG		AN	G				LG	Cap Gain LG/G	Prod Assu Appl	Grow Succ Pot	Prod Val	Total
G1-1	1.0	30	01	32	Kinetics of Nucleation and Crystal Growth	Zero Convection Zero Segregation	• •	• •	Liquid/Solid Interface Effects Homogen. Nucleation	o o	• •	G2	Thermal Gradient Contr. Supercooling		2	3	2	2	-	2.5
G1-2	1.0	30	01	32	Liquid/Solid Transition in Zero-G	Zero Convection Zero Segregation	• •	• •	Liquid/Solid Interface Effects	o o	• •	G2	Thermal Gradient Control		2	2	2	1	-	2
G2	1.0	-	01	32	Interaction of Surface Tension, Cohesion and Adhesion	Undisturbed Action of Intermolecular Forces	o	•	Liquid Shape Contr.	o	•		Surface Energies		3	3	2	3	-	3
G3	1.0	-	01	34	Zero-G Fluid Mechanics (Liquids, Mixtures)	No External G-Force Except G-Gradient Zero-Segregation	o •	• •	Acceleration of Liquids and Mixtures Flow Decay	o o	• •		Uniform Temp. Control Induced Liquid Motion-Techniques		3	3	3	2	-	3
H1	1.0	-	01	-	Mixing of Solids	Zero Segregation	o	•	Adhesion		•	G2 G3	Mixing Techniques		3	3	2	3	-	3
H2	1.0	-	01	32	Liquid/Solids, Liq./Liq. and Liq./Gas Mixing	Zero Sedimentation Mixture Stability	o •	• •	Interface Effects Mobility of Solids in Liquids		• •	G2	Mixing Modes Mixing Techniques		3	3	2	3	-	3
H3	1.0	-	01	20	Purification: Removal of Gases From Liquids	Mixture Stability	•	•	Interface Effects Marangoni Effect	• o	• •	G2	Thermal Gradient Contr. Induced Motion in Liquids	G3	2	3	1	1	-	2
H4	1.0	-	03	32	Position Control and Contact-Free Agitation	Contact-Free Suspension	o	•	Deformation/Oscillation of Liquids		o	•	Induced G-Forces Techniques		3	3	3	3	-	3
H5	1.0	-	01	-	Compaction	(Vacuum) Zero-Segregation			Particle Interface Effects	o	•	G2	Compaction Techniques Particle Preparation Vacuum Processing		2	2	3	1	-	2
H6	1.0	-	01	34	Casting Techniques	Mixture Stability Weightlessness			Interface Effects Flow Patterns	o	• •	G2 G3			3	3	2	2	-	2.5

1 = LOWEST  
3 = HIGHEST

• CONCLUSIVE VERIFICATION  
o DEMONSTRATION OR PARTIAL VERIFICATION

\* M. S. F. C. CLASSIFICATION SYSTEM

Table 3. Evaluation/Selection of Processes

Processes/Products (Abbreviated Designation)	Verification		Evaluation		Priority		Low-g Verif		Verification Material (prelim.)		Measurements	
	Anal.	1-g	Poten- tial†	State of Art†	1	2	0	Part Proc	Compl Proc	Sample Size*	Effect	Props uct
A1-1 Fiber/Particle Composite - Compact		0	2.5	3	6	1			•	3		•
A1-2 Fiber/Particle Composite - Mixing		0	2.5	2	8	1			•	3		•
A2-1 Controlled Density Met. - Compact Foaming		0	3	3	6	1			•	3		•
A2-2 Controlled Density Met. - Gas Injection		•	3	1	8	1		•		2	•	
A2-3 Controlled Density Met. - Ultrasonic Foaming.		0	2	0	10	2			0	2	0	
A2-4 Controlled Density Met. - Nucleate Foaming.		0	2	0	6	2			0	2	0	
A3-1 Unidirectional Eutectics	0	•	2.5	2	8	1			•	2	•	
A4-1 Supersaturated Alloys - Thermal Dispersion	0	•	2.5	2	0.1	1			•	1	•	
Supersaturated Alloys - Homogenization		•	3	1	8	1			•	2		
B1-1 Contact-Free Alloying		•	2.5	1	10	2		0	•	1/2	•	
B2-1 Superconductors - Predispersed Compact		•	3	1	10	2			0	2	•	
B2-2 Superconductors - Induction Mixing		•	3	1	10	2			0	2	•	
B3-1 Thermosetting Alloys	0	•	1.5	0	8		0					
C1-1 Single Crystals - Aqueous Solution		•	2	3	>100		0					
C1-2 Single Crystals - Molten Solution		•	2.5	3	>100		0					
C2-1 Single Crystals - Zone Melting		0	2.5	3	8	1			•	2	•	
C2-2 Single Crystals - From Melt		0	3	1	10	2			0	1/2	0	
C2-3 Single Crystal Wires/Ribbons		•	2.5	3	5	2			0	1/2		•
C3-1 Single Crystals - From Vapor		0	3	2	30		0		•	1		
D1-1 Amorphous Oxides (Glasses) - Preparation		•	3	2	10	2						•
D1-2 Amorphous Oxides (Glasses) - Shaping		•	2	0	30		0					

• = CONCLUSIVE VERIFICATION  
 0 = PARTIAL VERIFICATION OF  
 EVALUATION OF INDIVIDUAL  
 PROCESS PARAMETERS

\* (for definition,  
 see Sect. 5)

Table 3. Evaluation/Selection of Processes (Contd)

Processes/Products (Abbreviated Designation)	Verification		Evaluation		Priority		Low-g Verif		Verification Material (prelim.)	Measurements				
	Anal.	1-g	Poten- tial†	State of Art†	Min. Time (min)	1	2	0		Part Proc	Compl Proc	Sample Size*	Effect	Props
E1-1 Electrophoresis/Biochemicals/Stationary		•	2.5	3	10	1				•	1	Hemoglob./BPB	•	•
E1-2 Electrophoresis/Biochemicals/Continuous		•	2	3	>10			0						
E1-3 Electro-Magneto-Phoresis/Biochemicals	o	•	3	3	10	1				•	1	Urokinase Na, B	•	•
E2-1 Electrophoretic Separation/Isotopes		•	2	2	10		2							
E2-2 Centrifugal Separation of Isotopes		•	1.5	0				0						
F1 Drawing of Filaments	o	•	2.5	1	0.1	1				•	1	Metals, Oxides		•
F2 Growing of Whiskers			1.5	2	30			0						
F3 Drawing of Membranes	o	•	2.5	1	0.2	1				•	1	Sn, Ni	o	•
F4 Hollow Spheres		•	2	1	0.2		2			•	3	Al, Fe-alloy		•
F5 Blow Molding		•	1.5	2	4		2			•	3	Sn, Al	•	•
F6 Adhesion Casting		•	1.5	1	4			0						
F7 Slip Casting	o	•	1.5	3	20			0						
F8 Shapes by Deposition From Vapor/Mist		•	2	1	20			0						
F9 Grain Refinement Castings	o	•	1.5	1	8			0						
G1-1 Kinetics of Nucleation/Crystal Growth	o	•	2.5	3	5	1			•		2	Various	•	
G1-2 Liquid/Solid Transition	•	•	2	2	5		2		•		2	Various	•	
G2 Interaction/Surf. Tens./Cohesion/Adhesion	o	•	3	1	0.2	1			•		2	Various	•	•
G3 Zero-g Fluid Mechanics	•	•	3	2	6		2		•		3	Various	•	
H1 Mixing of Solids (Dry Mixing)		•	3	2	-			0						
H2 Liquid/Solid and Liq/Liq. Mixing	o	•	3	2	6	1				•	3	Al base	•	o
H3 Removal of Gases From Liquids	o	•	2	1	8		2			•	3	Sn, Al	•	o
H4 Position Control/Induction Mixing	o	•	3	2	4	1				•	2	Al, Fe	•	
H5 Compaction		•	2	3	-			0						
H6 Zero-g Casting Techniques		•	2.5	1	8		2			•	3	Various	•	

\* (for definition, see Sect. 5)

• = CONCLUSIVE VERIFICATION  
o = PARTIAL VERIFICATION OF EVALUATION OF INDIVIDUAL PROCESS PARAMETERS

† 1 = LOWEST  
3 = HIGHEST

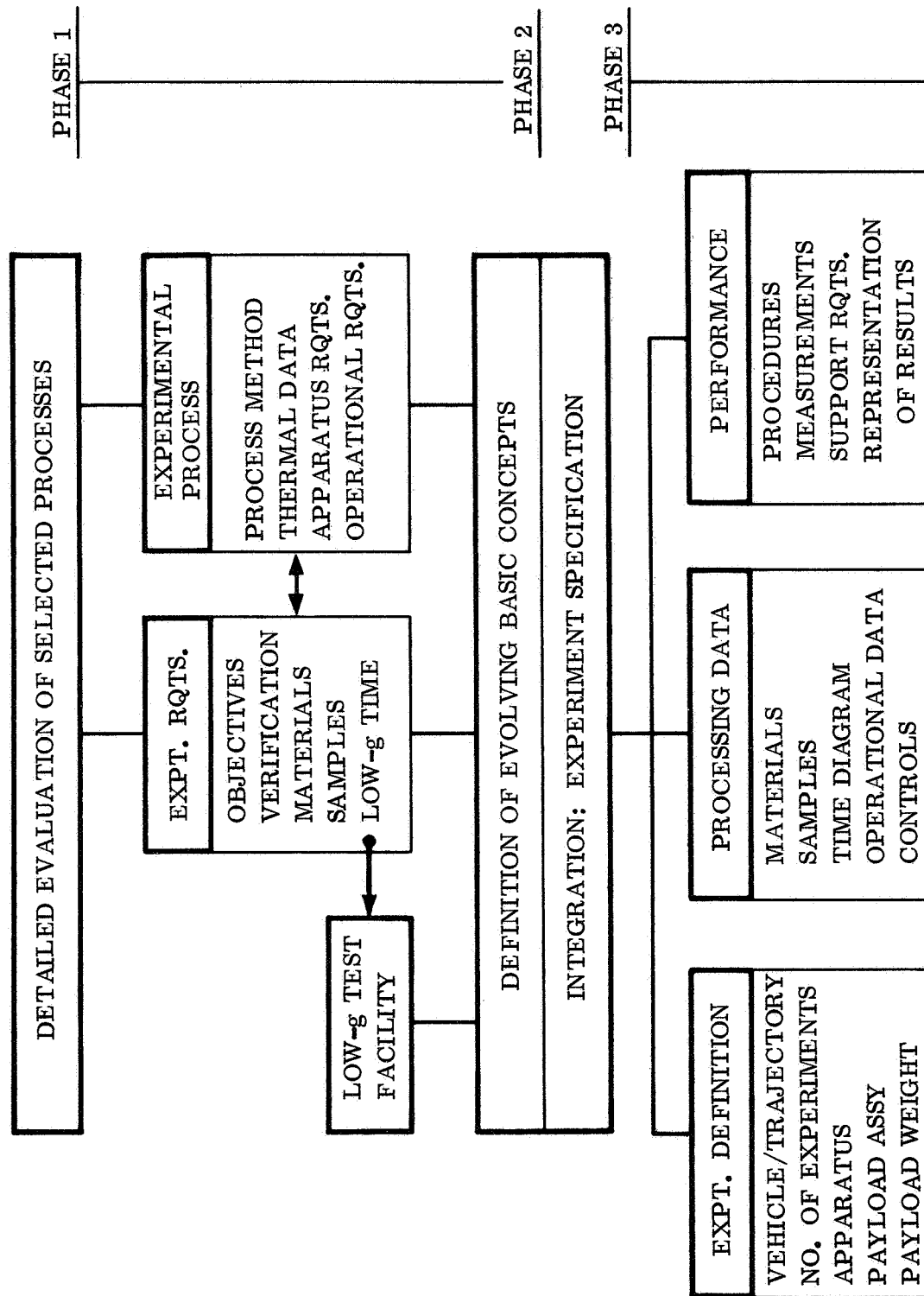


Figure 2-1. Task II Experiment Development and Specification

### 3. ADOPTED BASIC CONCEPTS

The present report is the result of several iterative evaluations. In earlier studies a number of basic approaches and concepts evolved which were then adopted as ground-rules and applied to all experiment definitions. It was seen fit to preface the report by a summary of the more significant adopted concepts. The following statements may serve as a rationale for the general approach and the selected experiment designs.

#### 1. Emphasis of Typical Experiments

Many processes, even though different in objectives and process classification, exhibit a high degree of commonality as to experiment requirements. It was attempted to narrow-down the multitude of specific experiments to a limited number of basic experiment types and apparatus designs adaptable to a wide variety of processes, even though different in nature and objectives. It is expected that the defined experiment types will also accept new, not yet defined processes, as they may evolve from the continuing process research.

Consequently, experiments representing basic types are discussed in greater detail, while the discussion of processes with similar experiment requirements and adaptable to a similar apparatus design are confined to the definition of specifics as to materials, processing parameters and product characteristics to be verified.

The resulting minimum number of apparatus types, covering a wide range of experiments, is in line with the objective of an experiment program of minimum cost and highest flexibility. Specific experiment requirements are satisfied by the modular apparatus concept, below.

#### 2. Verification Levels

In the objectives of individual experiments a distinction is made, wherever possible, between two verification levels, one for low-cost, near-term experiments for feasibility verification or for the establishment of experiences for more sophisticated experiments, and one generating conclusive process



and product capability data. This increases the choices in experiment programming and an adaptation to fluctuations in available funds. Often the verification levels also represent different low-g times and consequently the choice of tower or flight experiments.

### 3. Selection of Specific Materials

In most processes, the variety of experiment materials and possible sample quantities would result in a wide range of experiment requirements data. To arrive at more definite specification, data are computed for one carefully selected specific material, representing either the most severe conditions or a typical average condition. This is in line with the apparatus point design concept, discussed below.

### 4. Apparatus Point Design

Each experiment can be satisfied by a variety of apparatus designs. While many possible design concepts have been evaluated, one specific design concept has been selected in order to arrive at definite data as to dimensions, weight and operational requirements. The adoption of this "point design" concept is necessary to enable the definition of specific payloads within the limitations of zero-g facilities.

### 5. Multiple Flight Experiments

For tower experiments, the performance of one single experiment per drop is preferable to minimize apparatus cost and complexity; the performance of a series of experiments by a series of drops is acceptable in view of the comparatively low cost of tower experiments.

For flight experiments (KC-135, rockets) the opposite approach has been adopted: to achieve a high cost effectiveness and, at the same time, a high probability of success, each flight should carry the highest possible number of individual experiments, either of the same process with variations of materials or processing parameters, or "mixed" payloads consisting of individual experiments of two or more basic processes.

## 6. Modular Apparatus Design

For most experiments it was found advantageous to introduce a modular apparatus design. Rather than to place all samples in one single, large apparatus, each sample is processed in an individual apparatus ("processing module") which includes all direct support functions, such as heating and cooling, independent of other samples. The modular apparatus concept has numerous advantages (no negative points could be identified), such as:

- Each sample can be processed under different conditions (processing temperature and, consequently, material choice, heating profile, processing time and other processing parameters).
- There is no interference between samples with regard to outgassing and other high-temperature effects, as would be the case in a single chamber.
- The modular concept permits mixed payloads, with a wide choice of experiment combinations.
- Apparatus simplicity - as opposed to the necessarily more complex sample - chamber apparatus - and, consequently, higher reliability.
- High probability of test (flight) success: for instance, in the case of one malfunction only one experiment (sample) is lost; in the integrated apparatus, the entire test (flight) would be a failure.
- Individual modules are easy to install and to exchange.
- Fabrication of a series of identical modules is economical.
- Modules are convenient for developmental ground experiments.
- Use of one module for single-sample drop tower experiments.

## 7. Samples in Min. -g Position

The apparatus should be arranged so that all samples are at the position of

min. -g loads, i. e. in the longitudinal rocket axis. With the modular apparatus concept this is conveniently achieved by the "stacking" of processing modules along the payload axis.

#### 8. Basic Equipment Module

There is a considerable number of support equipment requirements which are common to practically all experiments. For rocket experiments it was, therefore, found technically and economically expedient to separate the payload into a basic equipment module and the processing apparatus. The basic equipment module provides the support functions for all experiments and is designed so that it accepts all types of processing apparatus. It consists of the following major components:

Rocket interface structure

Stabilization system

Payload "Can"

Batteries

Power conditioning

Timer and controls

Recorder

As a separate entity, the basic equipment module can be developed, fabricated and checked out independently of experiment development programs.

#### 9. Minimize Mechanical Actuators

In all apparatus designs, the use of active mechanical devices should be avoided or minimized. Mechanical actuator systems are complex, voluminous and unreliable. Wherever possible, easily controllable electrical, hydraulic or pneumatic systems should be used.

#### 10. Minimize Ground Support

It is advantageous, from the viewpoint of design and operations, to minimize ground support requirements and ground connections, prior and during test (flight). This includes such items as:

- Pre-launch ground support should involve only electrical connections (power, measuring, controls). Fluid connections are difficult to separate at launch time.
- The supply of all fluids (coolants, gases) and, wherever possible, electrical power should be integrated in the payload. This applies even to power supply for pre-launch pre-heating, since the battery weight penalty is surprisingly small.
- In-flight measurements are preferably recorded by a recorder which is installed in the payload "can." Telemetry should be limited to trajectory data, since transfer of signals from the stabilized payload can to the telemetry vehicle section (sliprings) is complex and unreliable.

#### 11. Rocket Point Trajectories

Each rocket can be fired at a wide variety of trajectories and, consequently combinations of low-g time and payload capability. For this evaluation only a few typical "point trajectories" have been used for each rocket class, in most cases the trajectories representing the min. and max. low-g time for a specific range (e. g. min. and max. White Sands Missile Range).

## 4. LOW-G TEST FACILITIES

Existing low-g test facilities applicable to developing space processing technology are of three types: Drop Tower, KC135 Research Aircraft and Suborbital Research Rockets.

### 4.1 DROP TOWERS AND AIRCRAFT

Drop towers and the KC135 Research Aircraft (Keplerian trajectory) are valuable tools in the study of low-g phenomena and development of process parameters. Only in isolated cases can they be adapted to exploratory experiments representing a complete process cycle (low-g alloying). Low-g times range from two seconds in drop towers to approximately eight seconds in aircraft. Capabilities of the MSFC drop tower, used as a model facility in this study, are summarized in Figure 4-1.

### 4.2 RESEARCH ROCKETS

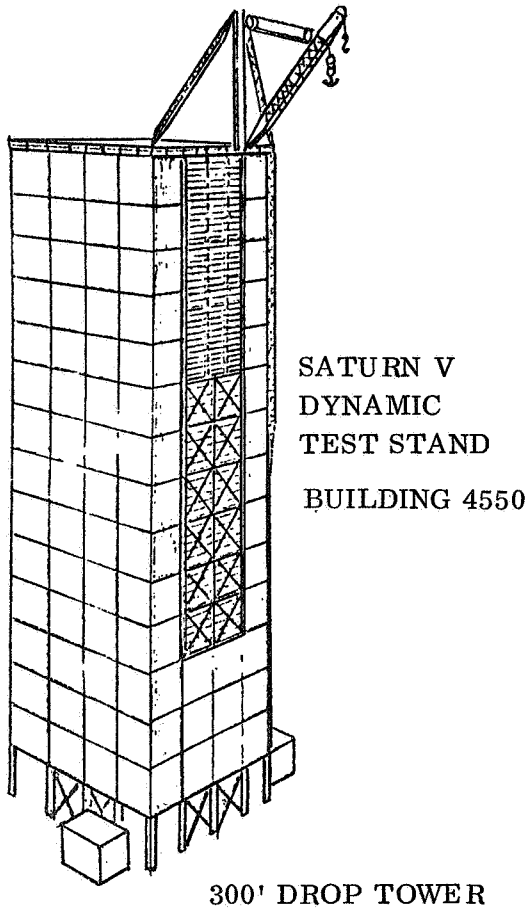
In this study, the national inventory of research rockets and their payload capabilities constitute the framework for the extended low-g experimental program. Only flights dedicated exclusively to space processing applications (SPA) are considered.

#### 4.2.1 Rocket Inventory and Capabilities

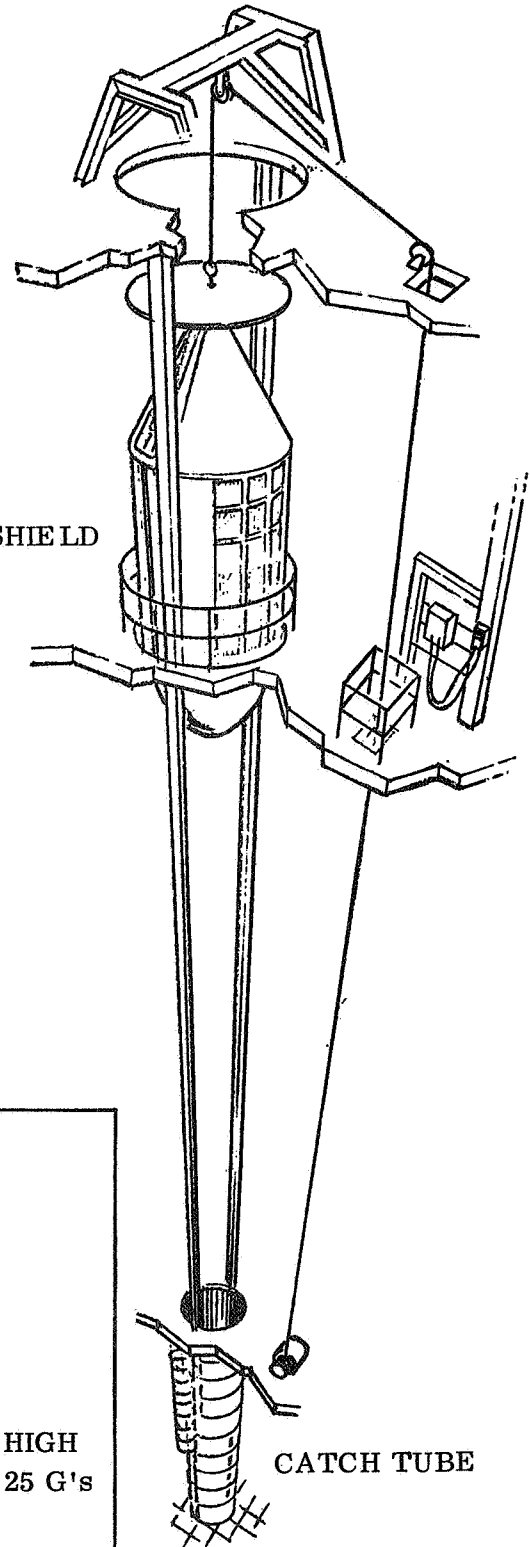
Substantial cost savings (without compromise of technical objectives) are available to the experimenter/mission planner by using existing vehicles rather than special orders, as this approach takes advantage of volume procurement. Rockets are obtainable from the manufacturers and/or government agencies listed below:

Black Brant - Bristol Aerospace Limited Winnipeg, Canada

Aerobee, Astrobee - Space General Company, Sacramento, California



DRAG SHIELD



#### CAPABILITIES

PAYLOAD	
PRESENT _____	450 LBS.
FUTURE _____	1000 LBS.
LOW GRAVITY TEST RANGE	
MINIMUM _____	$10^{-5} G_0$
MAXIMUM _____	$4 \times 10^{-2} G_0$
DROP TIME (295') _____	4.135 SEC.
TOTAL DROP WEIGHT _____	4000 LBS.
MAXIMUM TEST PACKAGE _____	3' DIA. $\times$ 3' HIGH
DECELERATION _____	LESS THAN 25 G's
INSTRUMENTATION CHANNELS _____	6
NON-DESTRUCTIVE TESTING	
ZERO TURN-AROUND TIME	

Source: MSFC - S&E - PE

Figure 4-1. The MSFC 300 Foot Drop Tower

\*NASA Goddard - Sounding Rocket Division (Karl Medrow)

\*USAF Cambridge Research Laboratory, Bedford, Mass. (P. Gustofsen)

\*Black Brant and Aerobee-Astrobee

Both manufacturers and Goddard were contacted during the study. Details of vehicle capabilities and launch sites were reviewed at length in order to provide a basis of trade-offs used in experiment design concepts. Data obtained are compiled in Table 4.1, and vehicle geometries are shown in Figure 4-2. Highlights of the data are as follows:

- Payload data (weight and envelope) are conservative approximations.
- Payload weight is defined as the total of experiment equipment and the rocket case extension which houses the experiment.
- WSMR Low-g flight times are limited by range size and the inaccuracies of vehicle flight trajectory caused by launch and weather variables.
- WSMR extended range costs more to use than the standard range. It is peopled and they must be evacuated during use. NASA-Goddard does not plan to use it.
- The exclusion of rockets from particular ranges serves the best interests of rocket selection for purposes of economy, range constraints and payload capability utilization.
- Costs shown in Table 4-1 are for flyable rocket motors only, exclusive of all hardware (payload) located forward of the motor case. The payloads have so many variables as to make cost generalities misleading.

The study shows that rocket capability exceeds requirements of the planned extended low-g experiments in terms of payload and Low-g flight times. Payload capabilities of the Aerobee 170 and 200 are sufficient for particular experiments, which in turn are tailored to these vehicles. Also both vehicles are on inventory. Aerobee 200

Table 4-1. Research Rockets and Capabilities for MS/MS Extended  
Low-g Experiments

Payloads and Launch Ranges								
Class	Vehicle	Payload Envelope Dia. & L. cm	***	WSMR Standard		WSMR**	Wallops/	Cost of Rocket *
			A-243 Sec Kg max	B-390 Sec Kg max	Extended C Kg max/sec	Churchill D Kg max/sec		
1	AEROBEE 170	38 X 228	130	86	75 / 427	N/A	42 K	
2	BLACK BRANT VC	41 X 228	318	210	136 / 508	136 / 493	37 K	
3	BB - NIKE VC	41 X 228	N/A	N/A	202 / 530	136 / 600	47 K	
4	AEROBEE 200	38 X 228	225	125	95 / 588	N/A	43 K	
5	AEROBEE 350	55 X 254	395	305	57 / 530	N/A	180 K	
6	ASTROBEE "F"	38 X 381	225	125	95 / 588	N/A	43 K	
7	MOD. ASTROBEE 1500	78 X 304	N/A	N/A	N/A	450 / 1200	200 K	

\* Cost for rocket engine(s) section only, excludes all hardware fwd. of rocket motors.  
For Example: These are "ready to fly" costs, plus the addition of an aerodynamic nose fairing (ogive).

\*\* Information only. Not planned for use by NASA - Goddard due to many operational problems. Also not needed for this program.

\*\*\* Best range for the program. Low operations cost and exceeds technical requirements.





has the largest current inventory, including unassigned vehicles and is most available. Another rocket, the Astrobee F, is attractive for its payload envelope and competitive cost. However, none exists presently, and it is doubtful that it will be available during the experiment program time frame. The first production order of 14 will begin delivery this (1973) summer.

One major technical problem of all rockets is that they impose an unacceptable g-load due to centrifugal forces of spinning and precession, the latter resulting from despinning. By contrast, the natural forces of the free fall ballistic trajectory, including aerodynamic drag are sufficiently small as to be ignored. This problem of rocket-induced g forces is not addressed in this study. Rather, NASA is seeking solutions to the problem as a separate activity.

#### 4.2.2 Costs of Rockets/Operations

Low cost, coupled with development goals, is an essential driver for achieving the proposed experiment program.

Since the Aerobee 200 is judged to be basic to the experiment program, cost information was obtained during the study and is presented to serve as an aid to the experimenter/mission planner. Aerobee 200 has ample payload capacity and costs are minimal. Significantly, the Aerobee 200 has a demonstrated re-use capability for two flights, and three flights are considered feasible. Costs for each re-use flight (vehicle only) are \$40,000 less than the price of a new rocket. In Table 4-2, costs are categorized as:

1. Rocket vehicle, exclusive of experiment package
2. \*Recurring operations consisting of:
  - a. Project or rocket field support. An experiment- sponsoring agency - Goddard interface to ready the rocket for flight, provide computer runs and flight analyses.
  - b. Base support. A Navy function, services such as facilities for building up rocket, flight scheduling, logistics and warehousing. Actual rocket firing is performed by the Navy.

- c. Range support. An Army function for services which include tracking radar, communications and telemetry receiving.

\*Assuming launch at White Sands Missile Range

Item Description	New Vehicle	Reuse Vehicle
1. Aerobee Rocket - Complete vehicle except for ACS, instrumentation and experimental package. Includes barrel extension to house experiment.	\$50K	10K
a. Support instrumentation: Telemetry, batteries, antenna, transmitter, timers, transducers, etc.	≈10/15K (a reuseable item)	None
b. ACS not needed, based on current planning to use a spin stabilized platform.		
2. Recurring Operations (for each flight)		
a. Project support	3K	3K
b. Base support	3K	3K
c. Range support (currently no cost to experimenter. Tentative for future)	3K	3K
Total Costs	69/74K	19K

Table 4-2. Aerobee 200 Cost of Vehicle/Operations

## 5. EXPERIMENT PAYLOAD PACKAGES

### 5.1 TOWER AND AIRCRAFT PAYLOAD PACKAGE

For drop-tower and KC-135 experiments, a basic payload package has been developed by MSFC whose operational capability has been proven in previous experiments. It has been found fully adaptable to all evaluated experiments. Fig. 5-1 shows the package with an installed experiment apparatus (gas injection foaming apparatus) as it is presently used in droptower and KC-135 experiments.

### 5.2 ROCKET PAYLOAD PACKAGE

For the payload requirements for low-g experiments in rockets, no precedent is in existence. Several "piggy-back" rocket experiments have been carried out by MSFC; however, the concerned payload was necessarily limited to a small single-experiment apparatus. For a high program effectiveness, piggy-back experiments are inadequate and the present evaluation is based on exclusive space manufacturing missions. This, in turn, calls for maximum utilization of the payload capabilities of the available rockets, discussed in Section 4.0.

The requirements which form the basis for the payload layout may be divided into two major groups: (a) the specific experiment (apparatus) requirements and (b) the experiment support requirements.

The basic approach to the rocket payload design was discussed in Section 3.0. There, the following basic concepts were adopted:

- (1) The payload is divided into two major subassemblies: (a) the experiment apparatus and (b) the support equipment.
- (2) Each flight should carry the maximum possible number of individual experiments (samples). Each experiment should be independent of others, which is, in most cases, satisfied by the modular apparatus concept.

- (3) In view of high commonality and recurrency, the experiment support requirements are most effectively combined in a standardized support module which satisfies the mechanical and operational support requirements of all experiments.

The following discussion defines (1) the major functions and components of the Support Module and (2) the support module design.

#### 5.2.1 Functions of the Rocket Support Module

The major functions of the Support Module are:

- (1) To satisfy the interface requirements between payload and vehicle.
- (2) To provide separable ground-payload connections
- (3) To provide experiment stabilization against rocket spin stabilization.
- (4) To provide structural support for the apparatus.
- (5) To provide power supply and power conditioning.
- (6) To provide first order controls and sequencing of events.
- (7) To provide means for in-flight recording of measurements.

The inclusion of the supply of expendables, such as gases or coolants, in the support module is optional. A considerable number of experiments require argon which is preferably supplied from a central storage vessel; this gas supply system may remain installed for several flights in the support module, or re-installed for each flight as part of the apparatus assembly. The same applies to a central coolant supply system; in most cases, however, the coolant supply is integrated in each individual processing module.

#### 5.2.2 Components of the Rocket Support Module

To satisfy these functional requirements, the support module consists of the following major components and subassemblies:

##### (1) Stabilization System

It includes the lower base plate (structural payload/rocket interface), the

lower slip-ring assembly (ground connections) and the female ground support plug assembly. The stabilization system is based on air bearings and positioning of the payload can, Figure 5-3, by one-sided weight distribution. It includes the gas supply for the axial-load air bearing; the low radial loads are absorbed by a low-friction ball bearing. This mode of stabilization was selected tentatively and motivated by the extensive experience of MSFC and Convair in the application of air bearing systems. Another alternative for low-cost stabilization (in contrast to a gyro system) is a geo-magnetic servo system. The payload space and weight requirements are approximately the same for both systems, so that the open choice does not affect the defined apparatus designs. The only difference of the geo-magnetic system is a longer payload can and a smaller base-plate section.

(2) Upper Base Plate Assembly

It consists of the upper base plate, the upper radial-load ball bearing and - if applicable - a slip ring assembly for transfer of signals to the telemetry section and/or a gas/steam venting system.

(3) Payload Can

Basic structural assembly of the spin-independent payload section in the form of a cylindrical "can." It contains - and provides structural support for - all subsequently listed components. The cylindrical envelope is either a closed wall with access doors, or (preferably) an open structural framework.

(4) Batteries

The battery pack consists of one or more 28V-DC batteries which can be easily exchanged. An Ag-Zn battery (Yardney PM-3 cells) has been selected, as it combines low weight, voltage stability and limited re-charge capability adequate for repeated ground check-out tests.

(5) Power Conditioning System

The need and degree of power conditioning varies extensively in the type of experiments. The system is, therefore, composed of modular units which can be arranged for, and adapted to, specific requirements.

Individual units are:

- (A) Power distribution and controls
- (B) Solid state inverter
- (C) Transformer
- (D) Solid State Rectifier

Only for a limited number of experiments can the power be "taken directly from the battery" and only unit (A) is required. This applies primarily to experiments with exothermic heating which need only service power, and to some cases of resistance heating. The majority of experiments requires high amperage/low voltage AC (units A, B, C). A few experiments call for low amperage/high voltage DC (electrophoresis) and all units (A) through (D). In the payload assembly sketches of Section 7, the power conditioning system is in most cases shown - for the sake of simplicity - in form of a single box; the individual units can, however, be arranged differently if indicated by operational considerations or for the purpose of weight distribution.

(6) Timer (Sequencer)

The timer/sequencer is essentially a solid-state electromechanical device, capable of on/off control of 40 events. It has only a low-current switching capacity and activates relays, power controls, solenoid valves, measuring circuits etc. Different types may be installed for specific flight requirements.

(7) Recorder

As pointed out in Section 3, in view of the difficulty of transferring measurement signals from the stabilized payload can to the spinning rocket telemetry section, measurements are preferably recorded within the payload can.

A 24-channel tape recorder with a signal conditioning provision is adequate for all experiments. If necessary, a second recorder can be installed within the space and weight contingency of the support module.

Major data for these components and totals for several typical support module assemblies are listed in Table 5-1. Minor interface components are included in the contingency data.

#### 5.2.3 Rocket Support Module Assembly

The complete assembly of the basic payload module is identified in Fig. 5-2. All support components located inside of the payload can are positioned so that their combined center of gravity is off the rocket axis. They can be rearranged for specific experiment requirements or for increased shift of the center of gravity.

#### 5.2.4 Apparatus Integration

The entire upper portion of the payload can is available for the apparatus assembly. In the standard (point design) rocket the net space of this section is 30 diam x 80 cm, and the net volume  $57,000 \text{ cm}^3$  (57 liters).

#### 5.2.5 Rocket Payload Stabilization

As pointed out earlier, two methods for stabilization of the payload can against the spinning rocket are considered: (a) air-bearing suspension of the payload can and (2) geomagnetic stabilization of the payload can.

Both methods call for a suspension of the payload so that it can freely rotate about the rocket axis with a minimum of friction. Of prime concern is the axial bearing load, which is the product of the axial resultant of the can weight and the launch acceleration. During the launch phase it reaches a maximum in the order of 800 to 2000 kg depending on payload can weight which, in turn, is related to the selected rocket type and trajectory. The radial loads are relatively small and in the order of 70 to 175 kg.

In the air-bearing stabilization method (a), the axial loads are absorbed by an axial air bearing located inside of the vehicle-fixed stabilization system, while



the radial loads are absorbed by ball or roller bearings. Stabilization is achieved by uneven weight distribution in the payload can so that its center of gravity is off the rocket axis. As the rocket spinning rate increases, bearing friction may cause the can to rotate back and forth, or even to go into a slow rotation. The g-loads induced by these motions are, however, negligible as compared with the g-loads induced by the rocket spin.

In the geomagnetic stabilization method, both, axial and radial loads are absorbed by ball or roller bearings. Stabilization is achieved by a geomagnetic, north-seeking sensor and a servo system which counter-rotates the payload can against the rocket spin at the same spin rate

For this evaluation, the air-bearing method (a) has been selected as model stabilization system. Its function is illustrated in Fig. 5-3. If the geomagnetic system (b) is used instead, the configuration of the lower payload section is modified. Various arrangements are possible between the payload apparatus and the stabilization system as shown in Figure 5-4. The space and weight available for the apparatus and support components does not vary greatly among the different configurations. The net available space for experiments is shown blank. The support system is cross hatched and the stabilization system has the cross on it. The configuration selected is the first on the left of the diagram.

Table 5-1. Components of the Rocket Support Module

No.	Component/Subassembly	Envelope Dimensions (cm)	Envelope Volume (cm <sup>3</sup> )	Weight (kg)	Footnotes
1	Stabilization System, including Lower Base Plate Assembly	38 dia. x 25	22,500	19.5	(1)
2	Upper Base Plate Assembly, including Upper Bearing Assy.	38 dia. x 5	1,800	4.5	(2) (3)
3	Payload Can	32 dia. x 120	97,000	9.0	(3)
4	Battery Pack - 28 V - 110 W-hr	9 x 9 x 14	1,140	3.0	(4)
5	Power Conditioning				
5A	Controls & Distribution	10 x 16 x 6	9,600	2.5/3	(5)
5B	Inverter with Controls	10 x 16 x 6	9,600	3.5	(5)
5C	Transformer	10 x 16 x 12	19,200	3.5/6.0	(6)
5D	Rectifier	10 x 16 x 6	9,600	1.0	
6	Timer - Sequencer	8 x 10 x 12	960	1.5	
7	Recorder Incl. Signal Conditioning	18 x 10 x 6	1,080	2.5	
	Weight Contingency (Wiring, etc.)			2	
Weight of Typical Support Modules					
	Min. Module with 28V DC Power Supply			45	
	Average Module with AC Power Supply			54	(7)
	Electrophoresis Support - High Voltage DC			52.5	
	Max. Module (3 kW/AC - 48 Recording Channels)			60.5	

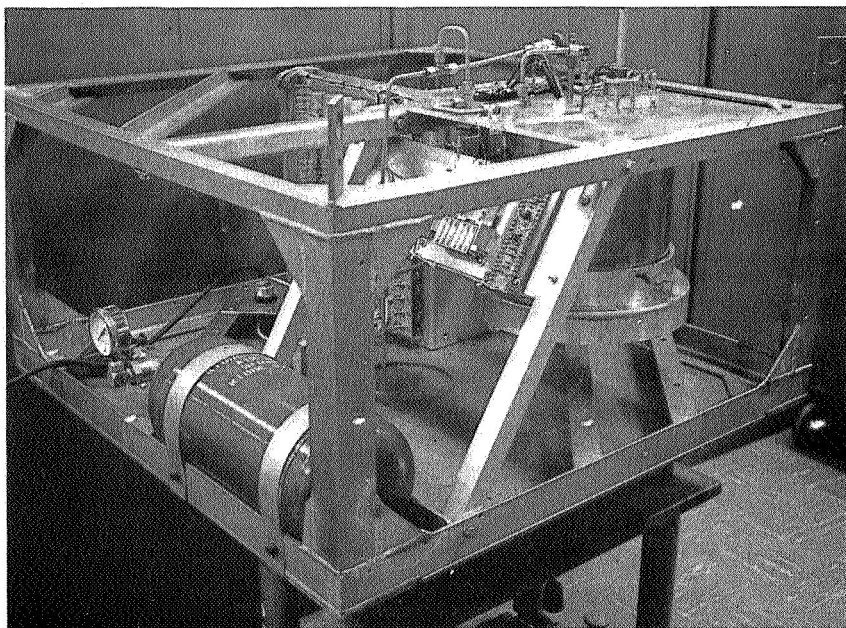
Footnotes:

- (1) Includes optional slipring assembly for 4 telemetry channels.
- (2) Includes slipring assembly for ground support circuits and female ground support plug assembly.
- (3) Includes gas - steam exhaust duct (through bearing center).
- (4) Max. discharge rate 60 Amps at 25 V (1500 W) each pack.
- (5) Weights 5A - 5B vary with placement of primary controls.
- (6) Two transformer types: 1 kW = 3.5 kg; 2 kW = 6 kg.
- (7) Weight range of average module: 51.5 - 57 kg.

Table 5-2. Typical Support Modules - Components and Weights

Table 5-2. Typical Support Modules - Components and Weights

No.	Component	Dimensions (cm)	Weight (kg)	Battery Direct		AC Power			DC Power		Electro- phones.	Max.
				1 kW	2.5 kW	1 kW	1.5 kW	3 kW	1 kW 200 W-hr	2 kW 300 W-hr		
1	Stabil. Syst./Base Plate	38 dia. × 25	19.5	x	x	x	x	x	x	x	x	x
2	Upper Base Plate Assy.	38 dia. × 5	4.5	x	x	x	x	x	x	x	x	x
3	Payload Can	32 dia. × 120	9.0	x	x	x	x	x	x	x	x	x
4	Battery(28 V)	9 × 9 × 14	3.0	x	xx	x	x	xx	xx	xxx	x	xx
5A	Power Distrib./Controls	10 × 16 × 6	2.5(3)	x	x	x	x	x	x	x	x	x
5B	Inverter with Controls	10 × 16 × 6	3.5			x	x	x	x	x	x	x
5C-1	Transformer	10 × 16 × 12	3.5			x					x	
5C-2	Transformer	10 × 16 × 12	6.0				x	x				x
5D	Rectifier	10 × 16 × 6	1.0									x
6	Timer-Sequencer	10 × 12 × 8	1.5	x	x	x	x	x	x	x	x	x
7	Recorder	10 × 18 × 6	2.5	x	x	x	x	x	x	x	x	xx
8	Contingency (Misc.)	-	2.0	x	x	x	x	x	x	x	x	x
1-3	Major Structural	} Sub Total		33	33	33	33	33	33	33	33	33
4	Battery Pack(s)			3	6	3	3	6	6	9	3	6
5	Power Conditioning			3	3	9.5	12	12	6	6	10.5	13
6-8	Timer/Recorder/Miscel.			6	6	6	6	6	6	6	6	8.5
Total Support Module Weights (kg)				45	48	51.5	54	57	51	54	52.5	60.5



**Figure 5-1. Drop Tower Package (MSFC)  
With Installed Apparatus (Gas Injection Foam-  
ing Experiments)**

## ROCKET PAYLOAD ASSEMBLY

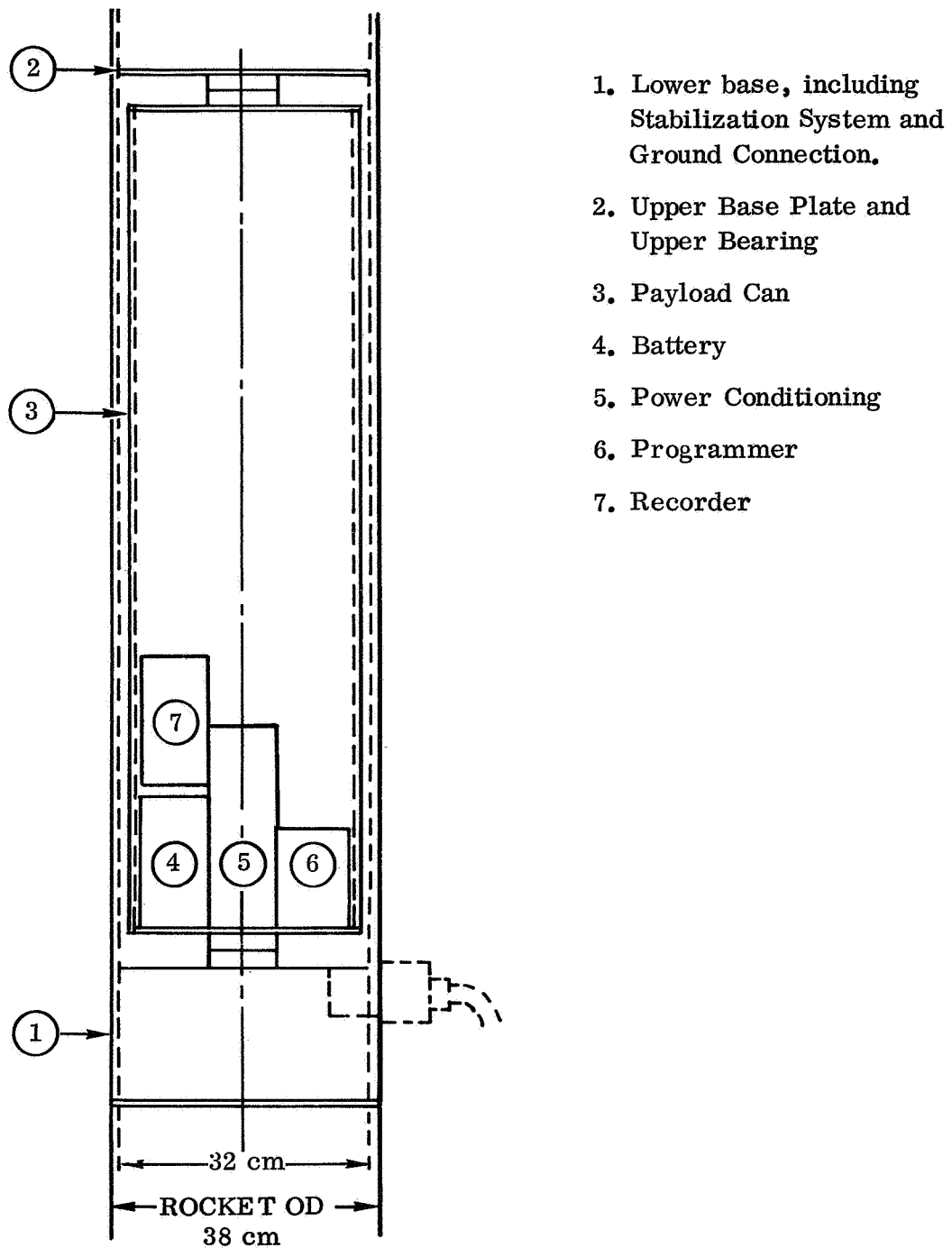


Figure 5-2. Rocket Experiment Support Module

## ROCKET PAYLOAD ASSEMBLY

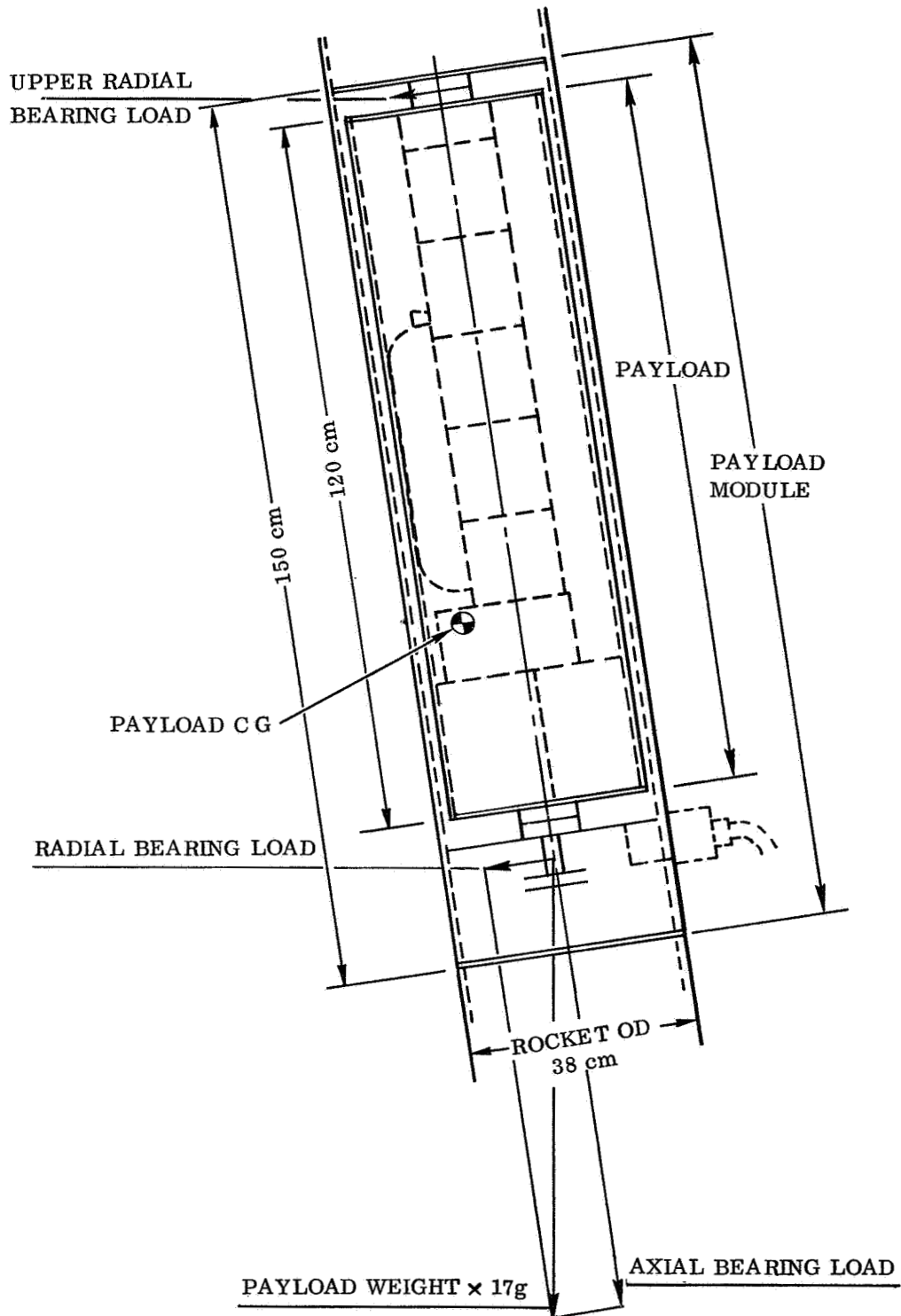


Figure 5-3. Air Bearing Payload Stabilization During Boost Phase



## 6. HEATING AND COOLING METHODS AND DEVICES

With the exception of material class I (biological materials), all processes involve heating and melting of the sample material. To minimize repetitious statements and figures in the individual experiment discussions, the reasoning for the selection of heating methods and the definition of heating systems are summarized in this separate section.

### 6.1 DISCUSSION AND SELECTION OF HEATING METHODS

An overview of the performance characteristics of various heating methods is shown in Fig. 6-1. An examination reveals that there are distinct differences as to specific characteristics, such as the methods of heat generation or heat transfer to the sample, the max. temperature capability, heating rate, time at processing temperature etc. From the viewpoint of the concerned experiments, the following requirements or characteristics are of primary significance for the selection of heating methods: 1) mode of sample suspension; 2) time at processing temperature and 3) the max. temperature. They are discussed in the following four sections.

#### 6.1.1 Mode of Sample Suspension

To introduce a clear terminology, typical suspension modes and the resulting material behavior during melting are identified in Fig. 6-2. In modes (1) and (2) the molten material is contained. In mode (2) the containment is achieved with a non-wetting (split) ceramic cover over the molten sample section, while accurate temperature (power input) control prevents extension of the liquid state beyond the cylindrical sample section. In modes (3) and (4) most of the liquid surface is contact-free and held in place either with stings (3) or by solid sample material (4). In the latter case, the liquid configuration becomes unstable at  $L = \pi D$ ; to assure shape retention, the length of the molten zone should not exceed  $2D$  (ratio of free to interface surface area = 4:1). In the "semi-free" resistance heating mode (5) the length of the melting



sample section of 4D is sufficient to assure instability and separation of the liquid, but short enough to produce a discreet, contact-free sphere. Since cooling sets in immediately upon separation and, consequently, power cut-off, this mode applies only to processes where a short melt cycle is adequate. Mode (6) represents contact-free suspension during melting and liquid state processing in an induction heating and position control system.

#### 6.1.2 Time at Processing Temperature

Considering the applicable modes of liquid material suspension, the heating methods can be classified with regard to liquid-state processing time as follows (time limits are typical, numbers in parentheses refer to sample suspension mode, Fig. 6-2).

##### Extended Liquid-state Processing Time - 30 Min.

- Radiation Heating/El. Res. Elements (1) (3)
- Direct Resistance Heating - Enclosed (2)
- Induction Heating - Molten Zone (4)
- Induction Heating - Free Processing System (6)

##### Intermediate Liquid-state Processing Time - 1 Min.

- Exothermic Heating (1) (3) (4)

##### Short Liquid-State Processing Time - 10 Sec.

- Direct Resistance Heating - Semi-free (5)

##### Very Short Liquid-state Processing Time - < 1 Sec.

- Electric Discharge Heating (5)

#### 6.1.3 Methods for Moderate Temperature

Moderate temperatures can be provided by all heating methods with the exception of electric discharge melting. This section discusses the methods which are most adaptable to the moderate temperature required. For electrical resistance heating

elements, the max. temperature is limited by oxidation of the conductor material and is, therefore, dependent on the environmental atmosphere. For most favorable conditions (min. heat loss), the max. temperatures of the heater and the sample for air and inert gas environment are as follows:

	<u>Air</u>	<u>Argon</u>
Typical Heater Materials	Ni-Cr,Cr-Al	Mo, W
Max. Heater Temp.	1300° C	>2000° C
Max. Sample Temp.	1000° C	1700° C

Considering secondary factors, such as the conductor suspension material and latitude in the furnace design/operation for initial experiments, the max. sample temperature for electrical heating elements has been placed at 1200° C. The sample temperature capability of the existing and previously flown MSFC rocket apparatus with a resistance furnace and conductive heat transfer is 400° C. Laboratory tests with improvised modifications carried out by Convair show that the capability can be increased to 700° C. Aluminum alloys have been successfully melted, even though the low wattage of the furnace requires considerable heating time and, consequently, ground pre-heating. The significant data for Al, derived from the recorded time-temperature diagrams are as follows:

Time to melting point	750 sec
Time for melting	420 sec
Water cooling through solidification	55 sec
Terminal water cooling to 130° C	130 sec

No test data and practical experience are available for the effectiveness of exothermic sample heating by radiation. On the basis of thermodynamic assessments, the max. attainable sample temperature - for short periods of time - is in the order of 1200-1500° C, depending on sample size. Considering secondary factors, such as exothermic container material, design simplicity and reliability, the max. temperature has also been placed at 1200° C.

#### 6.1.4 Methods for High Temperature

A substantial number of processes, particularly those concerned with alloying, call for extremely high temperatures in the regime from 1200 - 2500° C. In some cases, merely a melt-cycle is required; others call for control of heating rate, cooling rate, time at max. temperature or combinations thereof.

Various heating methods have been evaluated qualitatively and quantitatively. Most desirable would be induction heating in a free suspension system; it is, however not considered at this time because of the extensive equipment and control requirements and the undefined availability of an operational coil system. Conventional induction heating, which is the preferred laboratory technique for the concerned processing conditions, was found too cumbersome for flight experiments (equipment weight and volume, active cooling). Radiation heating with electrical resistance elements does not meet the temperature requirements, unless special techniques are introduced which would call for considerable development efforts. Electric discharge techniques may be applied in ground-based zero-g experiments where equipment weight and volume are of no concern. They are, however, only feasible for extremely small sample quantities using the "explosive wire" technique; the necessity of a wire of high L/D ratio results in undesirable end-shapes of inadequate size (multiple small spheres or odd-shaped pieces). Discharge techniques, finally, have the danger of excessive material vaporization. For these reasons, electric discharge techniques have been eliminated for flight experiments.

Detailed equipment design and performance studies of high-temperature heating techniques led to the clear choice of direct resistance heating as the method most adaptable to low-g experiments. It combines the following advantages: 1) capability of melting practically any metallic material; 2) adequate sample size, 3) adaptability to contact-free solidification, 4) adequate controllability, 5) accurate numerical definition of performance characteristics and the related equipment requirements, 6) simplicity and extensive use of off-the-shelf electrical components, 7) absolute reliability. It is described in detail in the following section.

### 6.1.5 Selection of Heating Methods

The net conclusion of the foregoing discussion is the selection of three basic heating methods for the initial (2-year) experiment program.

Radiation Heating with Electrical Resistance Elements

Exothermic Heating

Direct Resistance Heating

The rationale for the confinement to these three methods is the prime objective of the experiment program in the verification of processes and product capabilities. Experimental techniques and devices are of secondary concern; they should be effective and reliable, yet at the same time uncomplicated, to minimize time-consuming and expensive developmental efforts. Specifically, for all experiments which require heating, the choice of heating technique is immaterial as long as it provides the thermal characteristics needed to melt the experiment sample. The selected three methods satisfy the entire spectrum of heating requirements encountered in the defined experiments, as evidenced in the following chart (experiment requirements are identified by three basic conditions: temperature, time at temperature and mode of sample suspension; potential alternate methods in parenthesis).

Identification of heating methods:

ERE = Electrical Resistance Elements

RES = Direct Resistance Heating

EXO = Exothermic Heating

	<u>Sample Suspension</u>	<u>Short Time</u>	<u>Extended Time</u>
I:	<u>Max. Sample Temperature 1200° C</u>		
	Contained	EXO (RES-enclosed)	ERE
	Molten Zone	RES, ERE (EXO)	RES, ERE
	Min. Contact or Free	EXO, RES	(Not Required)
II:	<u>Max. Sample Temperature over 1200° C</u>		
	Contained	(RES-enclosed)	(Not Required)
	Molten Zone	RES	RES
	Min. Contact or Free	RES	(Not Required)

## 6.2 HEATING DEVICES - DESIGN AND OPERATIONAL DATA

### 6.2.1 Electric Radiation Furnace

Heating with electric resistance elements is preferred where accurate sample temperature control is required. Conduction heating by direct contact of the sample (container) with the heating element was eliminated in favor of radiative heat transfer for the following primary reasons:

- (1) Heat transfer conditions in a radiative arrangement are highly reproducible, while conductive heat transfer changes substantially with minute and unpredictable variations in the contact.
- (2) Need - in most cases - for a coolant passage between heater and sample.

The resistance elements consist either of sheet/foil or narrow-spaced wires, exposed to the environment. The use of filaments in quartz tubes is impractical in view of the necessary wide spacing and the incompatibility with water quenching.

On the basis of an evaluation of experiment requirements, three basic point designs for flat and cylindrical samples have been adopted:

6.2.1.1 The rectangular furnace for flat samples is shown in Fig. 6-3. Two heating panels with narrow-spaced heating wires are located between the sample and the walls of the rectangular processing chamber. For multiple experiments, individual furnace units are used which are stacked so that the samples are in the line of minimum g-level (e.g. rocket axis). This furnace is designed for a max. sample size of 2 x 2 x 0.5 cm. The performance data for a sample of this max. size, high sample heat content and a water-cooled chamber wall are:

Max. sample temp.	800° C	1200° C
Heater Temp.	1200° C	1525° C
Absorbed by chamber	700 Watts	1910 Watts
Absorbed by sample	550 Watts	640 Watts
Total power input	1250 Watts	2550 Watts
Heat-up time	37 Sec.	17 Sec.
Power consumption, net	13 Wh.	12 Wh.
- 28 V Battery, App.	0.5 Amph.	0.5 Amph.
Power rate to sustain max. temp.	400 Watts	900 Watts

6.2.1.2 The modular furnace for cylindrical samples is illustrated in Fig. 6-4. It is designed for the standard cylindrical sample of 1.2 cm diameter and 8 cm length. The heating element consists either of properly spaced coiled resistance wire (lower temperatures), or a split tungsten tube in an argon atmosphere (high temperatures). For terminal solidification, a closed active cooling system (see Section 6.3) is used. Following the principle of point-design data, performance data have been computed for the melting of aluminum:

Heating Element Temp.	1280	°C
Max Sample Temp.	700	°C
Heating Time, Solid State	76	Sec.
"    "    Melting	59	Sec.
"    "    Liquid State	5	Sec.
Total Heating Time	140	Sec.
Power Rate - Max.	1800	Watts
- to sustain processing temp.	500	Watts
Power Consumption - Heating	49.7	Wh.
- 60 sec. at processing temp.	8.4	Wh.
Total Power Consumption	58.1	Wh.
" - 28V Battery, App.	2.5	Amphrs.

The sample temperature profile for a heating element temperature of 1250° C is shown in Fig. 6-5A and the power (heat input) profile in Fig. 6-5B. The active cooling profile for the closed cooling system and various water flow rates is identified in Fig. 6-6.

A tubular furnace for multiple experiments under identical processing conditions is illustrated in Fig. 6-7. This particular version is designed for axially expanding samples (foaming), leaving space for expansion and for arresting in expanded position. It uses a split tubing as heating element and a closed cooling system for terminal solidification. For aluminum-base samples, the temperature profile and the times for heating and cooling are identical to those of the modular furnace (Figs. 6-5 and 6-6);

the power and coolant requirements can be approximated by multiplying the values of the modular furnace with the number of samples, since the fraction of the furnace height required for each sample is identical to the height of the modular furnace.

### 6.2.2 Exothermic Heating

Substantial amounts of heat can be generated by exothermic reaction of a suitable material. Space-rated exothermic materials with a reaction heat in the order of 600-800 cal/gr are available. The reaction temperature is in the order of 3000° C. While the reaction is instantaneous (less than one second), it can be slowed-down by the addition of an inactive material, such as glass powder, at the expense of the heat generated per unit of volume. The exothermic reaction transforms the material into a solid briquet which can be maintained at high temperature for considerable time by appropriate insulation of the system. During combustion a moderate amount of gas (app. 0.05 liters/gr) is generated which has to be vented from the system.

The basic design of the exothermic processing unit is shown in Fig. 6-8. Heat from the briquet is transferred to the sample preferably by radiation from the processing chamber wall. Direct conductive transfer to the sample is less desirable in view of the resulting high thermal gradient in the sample material. The sample cooling rate can be regulated by the amount of external insulation and heat radiated to the environment or a coolant. In limited-time experiments, terminal cooling by water injection into the processing chamber may be required. In orbital experiments, where time is not critical, extremely low cooling rates can be achieved.

The amount of heat transferred to the sample by radiation in the arrangement of Fig. 6-8' has been calculated for two point designs:

Unit I: 12 diam x 12 cm

Unit II: 10 diam x 10 cm

Most of the generated heat is absorbed by the metallic container. To keep its wall thickness as low as possible, all units have to be vented to preclude pressure build-up. Data for the two point designs, which cover essentially all experiment requirements are as follows:

### Exothermic Material Data

Type	Narmco Exotherm 34
Density	2.88 gr/cm <sup>3</sup>
Reaction heat	700 cal/gr (min. avg.)
Released gases	0.05 l/gr
Gas composition	95% H <sub>2</sub>
Reaction product	Solid Bricket

### Apparatus Data

	<u>Size I</u>	<u>Size II</u>
Diam. (less insul) (cm)	12	10
Length (less insul.) (cm)	12	10
Chamber diam (cm)	4	4
Chamber length (cm)	12	10
Wall thickness (cm)	0.2	0.2
Metal volume (cm <sup>3</sup> )	169	125
Exo. mat'l. vol. (cm <sup>3</sup> )	1200	650
Chamber volume (cm <sup>3</sup> )	150	125

### Performance Data

Max. chamber temp. (° C)	2500	2000
Total Exoth. heat (Kcal)	840	455
Heat loss/max. insul.) (Kcal)	473	340
*Net heat to chamber/Kcal)	363	115

\*Heat required (Kcal) to melt<sup>1)</sup>:

1.2 diam x 7 sample, Al	6.9
" " Ni	19.5
2 cm <sup>3</sup> containerless, Max.	7.4

1) including container and suspension

A comparison of the available and required heats (\*) shows that there is a high contingency, allowing temperature - time control by less insulation or by passive additions to the exothermic material.



The heating profile of the exothermic material is shown in Fig. 6-9a for max insulation (essentially no wall losses) and for deliberate partial heat transfer to the environment. The resulting temperature profiles for a containerless small sample ( $1-2 \text{ cm}^3$ ) are identified in Fig. 6-9b and for a contained sample of  $8 \text{ cm}^3$  in Fig. 9-c. The diagrams - to be verified by experiments - show that an appreciable temperature can be maintained over a time period in the order of 40 seconds for the highly insulated system.

The combination of limited operation time and operational simplicity makes exothermic heating particularly adaptable to rocket experiments. It is further highly adaptable to extravehicular orbital experiments where long cooling times at low heat transfer rates can be achieved. A typical design for extravehicular space experiments is shown in Fig. 6-10 which includes provision for chamber atmosphere control and for unit recovery after complete cooling and gas consumption.

### 6.2.3 Direct Resistance Heating

Sample heating by its own electrical resistance calls for currents between 200 and 500 amps. for the involved sample sizes. For tower experiments, where the short low-g time limits the sample size, 200 amps could be supplied by several single-cell batteries arranged parallel. In this case, the bulky and heavy battery, switches and leads are acceptable.

For rocket experiments, this current supply method is unfeasible, since the weight and size of switches and control components for currents in the order of 500 amps. are prohibitive. An inverter-transformer system proved to be very attractive for the following reasons:

- (1) Use of the standard 28-V flight battery.
- (2) All controls can be placed in the primary circuit, using standard control elements of small size and weight.
- (3) By use of individual transformers for each sample, they can be adapted exactly to the electrical characteristics of the sample material.
- (4) Lightweight (20 amp) wiring.

The electrical system, illustrated in Fig. 6 -11 consists of the battery, the central solid-state inverter (600-5000 Hz) with integrated controls and the transformer which is an integral part of the processing module. The conceptual design of the processing module is shown in Fig. 6-12. Its major subassemblies are

- (1) The processing chamber
- (2) The sample assembly
- (3) The transformer.

The processing chamber is a rectangular insulated container, pressurized with argon at 1-1.5 atm. The specially-built, yet inexpensive high-frequency transformers have a single-turn secondary winding to provide the high required current. The selection of amperage and frequency can be matched with the sample resistivity. The secondary turn is a U-shaped heavy copper bar. The sample assembly consists of the high-mass ends of the copper-U, in which the sample ends are embedded, closing the secondary turn. The approximate dimensions for drop-tower and rocket experiments are:

	<u>Tower Module</u>	<u>Rocket Module</u>
Max. Power Input	400W	1000W
Dimensional Envelope (cm)	14 x 16 x 18	14 x 16 x 18
App. Weight (Kg)	12	12

The basic sample configuration is shown in Fig. 6 -13A. Upon melting either of the following may occur:

- (a) The sample stays intact, center section slightly deformed by surface tension.
- (b) The center section separates and forms 2 semi-spherical samples (Fig. 6-13B)
- (c) The center section breaks apart into a free sphere and two end pieces as in (b) (Fig. 6 -13C)

If the sample is to stay intact (A), as in the case of crystal growth experiments, the L/D has to be less than  $\pi D$ , preferably only 2D. For contact-free solidification experiments, an L/D of 4 is most likely to produce the desired condition (C).

In accordance with the material quantities required for applicable experiments, two standard sample sizes have been selected: 0.2 cm diameter (tower experiments) and 0.4 cm diameter (rocket experiments). For condition(C) the exact sample dimensions before and after melting are as follows:

<u>Sample I</u>	<u>Original Sample</u>	<u>Resulting Evaluation Samples</u>		
Shape	Cylindrical	Sphere		Half-Sphere
Number	1	1	and	2
Diameter (cm)	0.2	0.25		0.3
Length (cm)	0.8	-		0.15
Volume (cm <sup>3</sup> )	0.025	0.008		0.007

<u>Sample II</u>	<u>Original Sample</u>	<u>Resulting Evaluation Samples</u>		
Shape	Cylindrical	Sphere		Half-Sphere
Number	1	1	and	2
Diameter (cm)	0.4	0.45		0.55
Length (cm)	1.6	-		0.3
Volume (cm <sup>3</sup> )	0.201	0.047		0.045

The power and equipment requirements for sample heating and melting are determined by the total thermal profile consisting of:

- (1) Heat absorbed by the heat content of the sample, i. e. solid-state heating and heat of fusion.
- (2) Heat losses by radiation.
- (3) Heat losses at the sample ends.

Extensive numerical trade-off studies were carried out to arrive at an optimum combination of time at the melting temperature, acceptable heat losses and acceptable power requirements.

For the short processing times of Sample I, the end-losses can almost be neglected, and the heat requirements are primarily composed of sample heat content and radiation losses. The resulting data are stated below.

For the extended time requirements of Sample II, most of the energy output is lost at the sample ends (app. 55-70% of the total input). This high loss led to the specific Sample II configuration, Fig. 6-13A, designed to reduce the end losses. Sample heating accounts for app. 10-15%, and radiation for 20-25% of the total heat.

An optimized processing profile for Sample II, which serves as model for the low-g time definition, is shown in Fig. 6-14. It is computed for the Nb-Al-base alloys with a melting temperature of 2200° C and is representative of most of the applicable high-temperature experiments.

The thermal profile (Fig. 6-14A) identifies a total heating time of 28 sec. The solidification time of the resulting free sphere is less than 2 seconds, placing the total processing time at 30 seconds. For some alloys, an optimum is obtained at a somewhat shorter or longer total time; a maximum total time of 40 seconds has, therefore been adopted as experiment base value. To assure that none of low-melting constituents, such as gallium, is lost, all this processing time should be under low-g conditions.

The heat/power profile (Fig. 6-14B) identifies a maximum power input of 700 watts. This can be easily achieved with the high frequency transformer (Fig. 6-12) and an output of 0.5 volts and 1400 amps. For each specific sample composition, the

frequency, voltage and amperage have to be matched with the sample resistivity. The extremely low total energy requirements of 6 wh (= 0.2 amp hrs for a 28-volt battery) do not warrant any preheating on the ground.

The maximum processing times and power requirements for samples I and II are summarized below

Sample	I	II
<u>Max. Time (seconds)</u>		
Heating/Melting	2.5	38
Solidification	0.5	2
Total Low-g	3	40
<u>Power/Heat</u>		
Max. Current (Amps)	300	1500
Max. Inputs (Watts)	400	1000
Total Energy (Wh)	0.25	6
Total Heat (Cal)	210	6000

### 6.3 COOLING METHODS AND DEVICES

In all experiments involving sample melting, the sole purpose of cooling is to attain complete sample solidification, while the remaining solid-state temperature is of no concern. In most cases the cooling rate is immaterial as long as the sample is reliably solidified at the end of the low-g period. Control of the cooling rate, prior to terminal solidification, is only required in processes involving crystal growth or directional solidification.

#### 6.3.1 Cooling Methods

As to the methods of cooling, we may distinguish between three modes: 1) natural (passive) cooling by radiation, 2) controlled cooling and 3) "terminal cooling" to assure complete solidification at the end of the low-g time.

The rate of natural (passive) cooling, essentially by radiation, depends on 1) the initial temperature, 2) the material mass, 3) the mode of sample suspension and 4) the design of the heating device. Consequently the time required for solidification varies extensively for specific combinations of these four conditions and ranges from 10 minutes for low temperatures and large contained samples to less than one second for high solidification temperatures and small, open samples. It is apparent that for passive solidification times which represent a substantial fraction of the low-g time or more, active terminal cooling is required to assure complete solidification within the low-g period. Several methods for terminal cooling could be considered; the use of water as coolant is preferred since it is efficient and comparatively uncomplicated. Means for the control of the solidification rate during low-g processing have to be designed individually for specific requirements and are integrated in the applicable processing apparatus.

The chart of Fig. 6 -15 identifies the modes of cooling applicable - as a rule - to various temperature levels and modes of sample containment or suspension. It is based on the sample quantities as they are predominant in each temperature regime and suspension mode. In the case of immiscible systems, the temperature level applies to the lowest melting constituent. The chart shows, that in the low temperature regime active terminal cooling is required in all cases. In the intermediate temperature range the necessity of active cooling depends on the specific combination of conditions. At high temperatures, solidification is, as a rule, accomplished by radiation.

The chart further identifies the adaptability of heating methods (in terms of temperature and sample suspension mode) to solidification rate control during processing.

### 6.3.2 Active Cooling Systems

From the viewpoint of functional concepts and equipment design, active cooling represents one of the most difficult problems of space processing systems since it is highly dependent on the environmental conditions peculiar to space operations. In contrast, heating and power systems are insensitive to the environment, so that terrestrial methods and

existing hardware can be used with only secondary modifications. This conclusion also evolved from prior space laboratory studies, even though its importance has not been generally recognized.

Typical problems of cooling systems for space manufacturing operations are the absence of a conductive environment, the complexity of radiative heat dissipation systems, the extremely high amounts of generated heat, the high heat peaks and the coolant management under zero-g conditions. Specific problems encountered in rocket experiments are

- (1) At the end of the zero-g time, all experimental material has to be returned to the solid state. In view of the zero-g time limitations of rocket flights, terminal cooling from high temperatures has to be accomplished within a very short time in the order of 30 seconds, representing extremely high cooling rates.
- (2) At these high cooling rates, the formation of coolant steam is unavoidable, or even necessary to achieve high cooling efficiency. This steam has to be either blown overboard or recondensed as quickly as possible to preclude excessive volume or pressure peaks in the cooling system.
- (3) All coolant and steam management has to be adapted to the varying g-levels and g-vectors of the rocket flight.
- (4) High cooling efficiency calls for intimate contact of the coolant with the hot surfaces. This, in turn, necessitates adequate coolant passages in the processing chamber, high coolant flow rate and high coolant turbulence to minimize film boiling.
- (5) The disposal of steam overboard is difficult due to the relative rotation of the payload can with regard to the rocket.
- (6) In some experiments it is necessary to continue cooling beyond the initial terminal cooling (solidification) period.

Since cooling and solidification is an equally important part of the processing cycle as heating and melting, extensive studies of cooling methods and systems were carried out; many originally adopted concepts were discarded in the course of these efforts in view of functional problems which were initially not recognized. Even though a discussion of these studies would serve as a rationale for the selected concepts, it is omitted as it

exceeds the scope of this report. Two basic cooling systems were adopted.

- (1) The "open" system
- (2) The "closed" system.

For both types, preference was given to the modular design in which each processing unit has its own independent cooling system for the reasons outlined in Section 3.6. While a central cooling system for multiple experiments is feasible, it is difficult to provide space in the payload can for a coolant tank of proper length to diameter ratio.

Several coolants were evaluated including liquid nitrogen. It was concluded that plain water is superior, considering all factors such as heat absorbing capability, boiling temperature, equipment requirements and simplicity of coolant management.

(1) The open system is illustrated in Fig. 6-16. While it is comparatively simple in design and operation, its application is limited to small processing chambers and to sample materials with a discreet solidification temperature. The coolant (water) is delivered to the hot surfaces from a pressurized supply tank by means of a spray system. Heat is absorbed primarily by coolant vaporization. Concurrently with the opening coolant valve, a vent valve opens, directing the steam into a central exhaust tubing. For overboard disposal, the steam is transferred from the payload can to the exhaust duct of the revolving rocket section through the hollow shaft of the upper bearing assembly. The cooling and water flow rate is primarily limited by the volume of steam which can be vented overboard per second. For the average size processing chamber of app.  $200 \text{ cm}^3$  volume, the following data apply:

Heat content of processing chamber, app.	48,000 cal
Water spray rate	4 cc/sec
Average heat absorption rate (varying steam/water ratio)	1,200 cal/sec
Total active cooling time	50 sec
Total water consumption	200 cc
Max steam exhaust rate	2.5 l/sec
Heat absorbed in 30 sec	39,000 cal
Heat absorbed in 50 sec	48,000 cal
Water reserve after 50 sec	200 cc



(2) In the closed system, Fig. 6-17, (this figure represents a functional diagram of the cooling system; in modular apparatus arrangements, coolant system and processing chamber are integrated in one unit, as illustrated in Fig. 6-4), coolant circulation is maintained by means of a constant-displacement pump, from the start of pre-launch operations throughout launch and flight to payload landing. At all times, the coolant is kept in rotation in the supply tank by tangential injection of the coolant return. This is necessary in order to assure contact of the pump with liquid coolant under all g-conditions. The pattern of coolant distribution under various g-conditions and flight phases is illustrated in the insert of Fig. 6-17. During the period prior to terminal cooling, all coolant flows through the by-pass line, while the processing chamber is cut-off by means of a control valve ('valve assembly'). For terminal cooling, the control valve directs a gradually increasing portion of the coolant into the processing chamber and back to the return line through a check valve.

During the active cooling period, the coolant is delivered into the processing chamber by means of an injection system with multiple injection (spray) elements. To minimize steam generation in the injection system, only the spray elements are exposed to the heat, while all coolant distribution elements are located outside of the insulated chamber.

As indicated in Fig. 6-17, the coolant volume is only one-half of the volume of the cooling system. The ullage is necessary to provide a "pillow" for the steam generated during the initial cooling period and to minimize pressure increase in the cooling system. Most of the steam will, however, recondense in the return line where it mixes with the coolant fraction still flowing through the by-pass. Any remaining steam joins the centered ullage in the supply tank, where it recondenses quickly. (This steam is responsible for the conical shape of the return-side ullage during cooling; at other low-g times the ullage is perfectly cylindrical).

To assure fast and complete steam recondensation, the total heat capacity of the coolant supply has to be sufficiently above the total heat stored in the processing chamber. The dimensional and thermodynamic data of the cooling system for a typical modular processing chamber and a sample temperature of 700° C (Al) are as follows:

Module dimensions (Fig. 6-4)	25 diam x 16	cm
Net volume of processing chamber	600	cm <sup>3</sup>
Coolant tank dimensions	20/24 diam x 12	cm
Cooling system volume	4	liter
Coolant supply	2	liter
Max heat capacity ( $\Delta T = 50^{\circ} \text{C}$ )	100,000	cal
Total power input (Sec. 6.2.1.2)	58.1	wh
Total stored heat	50,000	cal
Max. coolant flow rate in chamber	30	cc/sec
Time to cool sample to $90^{\circ} \text{C}$	80	sec
Coolant temperature increase	25	$^{\circ} \text{C}$
Reserve cooling capacity	50,000	cal

Using the full cooling capacity and allowing a coolant temperature increase of  $50^{\circ} \text{C}$ , this system can be used for sample temperatures up to  $1100^{\circ} \text{C}$ . For higher temperatures, the module size has to be increased.

For processing systems of lower heat content (small sample and chamber, moderate temperature) a simpler version of the closed system can be used, as it is shown in Fig. 6-3. In this arrangement, the tank is completely filled with coolant. Coolant circulation (and rotation in the supply tank) is initiated at the time of terminal cooling start/opening of control valve and maintained to the time of payload touch-down. Due to the coolant rotation, any steam entering the supply tank is forced toward the inner cylindrical tank wall, recondensing quickly during this process. The resulting transient and limited volume increase is absorbed by the expandable outer tank wall.

In all cooling systems, any atmosphere control of the processing chamber (such as maintaining argon atmosphere) is discontinued concurrently with the start of active cooling.

### 6.3.3 Cooling of Exothermic Furnaces

Exothermic heating is very attractive in view of its simplicity. However, cooling is difficult compared with other heating methods since the source of heat cannot be cut-off. In orbital operations this problem is less severe, particularly in extra-vehicular operations, as there is ample time for slow cooling by radiation. It is apparent that the limited low-g time of rocket experiments does not permit radiation cooling, particularly since all heat has to be contained in the furnace to preclude undue temperature rise in the payload module.

Numerous cooling concepts have been studied, such as

- (1) Separation of the sample from the continuously radiating chamber wall by removal of the sample or the furnace.
- (2) Separation of sample and chamber wall by insertion of a tubular passive heat shield.
- (3) Separation combined with active cooling by insertion of a water-cooled heat shield with or without water spray against the sample.
- (4) Injection into the chamber of a slurry which reacts endothermically and, at the same time, provides an insulation between sample and heat source.
- (5) Active cooling of the exothermic briquet in the region adjacent to the chamber wall.
- (6) Water injection from the chamber ends against sample and chamber wall (complete vaporization - "open" system).

Removal of the sample (1) is unacceptable due to the involved g-loads. Removal of the furnace was also eliminated in view of the high payload space penalty. In addition, both methods still leave the sample to inadequate cooling by radiation.

Method (2) is unfeasible, since it essentially traps the sample heat. The cooling effectiveness of method (4) was found inadequate, aside from the complexity of the system. Method (5) was eliminated due to excessive coolant and time requirements.

This left only methods (3) and (6). The insertion of a water-cooled heat shield with spray action toward the sample (3) was found most effective; it was however considered too complex for initial experiments and dropped - for the present time- in favor of a stationary injection system (6).

To be on the safe side, the net water supply volume for this "open" system is selected so that its total heat content (complete vaporization) is equal to the total exothermic heat. For the systems defined in Section 6.2.2 with a generated exothermic heat between 455,000 and 840,000 cal a net water supply of 0.75 to 1.4 liters is required. As illustrated in Fig. 6-8 (Sect. 6.2.2), the water is stored in a spherical container with an expulsion bladder and pressure-fed to the chamber; the exhaust steam is disposed overboard as described for the open system in Section 6.3.2.1. After cooling start, the system remains open, so that all water is consumed, partly during the terminal low-g time and partly during payload descent (parachute). The system is insensitive to g-loads or payload attitude.

During operation, the sample is solidified within less than 40 seconds and "kept cool" in the remaining cooling time (app. 240 sec). At the same time, the chamber wall and the adjacent portion of the briquet are cooled-down, cutting-off the heat transfer to the sample. It is expected that the cooled part of the briquet eventually serves as a ceramic insulation between the hot part and the chamber wall.

The major dimensional and thermodynamic data for the max. exothermic system (No. II - 840 kcal) are as follows:

Supply Tank Dimensions	15 cm I. D.	sphere
Tank Volume	1.75	liters
Water Volume	1.65	liters
Useful Water Volume	1.40	liters
Heat Absorption Capacity	840,000	cal
Total Exothermic Heat	840,000	cal
Water Injection Rate	5	cc/sec
Heat Absorption Rate	3,000	cal/sec
Total Operation Time	280	sec
Max. Heat Stored in Sample	20,000	cal
Time to Solidify Sample	<40	sec
Steam Exhaust Rate	5	lit/sec

These data are based on a conservative systems sizing in view of the many assumptions which have to be made in the theoretical thermodynamic assessment. Accurate data can only be obtained in ground or ground simulation experiments. It is expected that they will permit a substantial reduction of the systems size.

HEATING METHOD	HEAT GENERATION	SAMPLE SUSPENSION	HEAT TRANSFER	MAX. SAMPLE TEMP. (1)	HEATING RATE (2)	TIME AT MAX. TEMP (2)	PASSIVE COOLING RATE (2)	TEMPERATURE CONTROL	ADVANTAGES	DISADVANTAGES
A ELECTRICAL RESISTANCE ELEMENTS	Electrical Resistance (Indirect)	A-1 Contained	Radiation or Conduction	1200°C	Very Low	Unlimited	Low	Good	Sustained Processing Temperature	Limited Temp. Capability Slow Heat-up
		A-3 Sting		1100°C			High	Fair		
		A-4 Molten Zone		800°C			Medium	Fair		
B EXOTHERMIC HEATING	Reaction (Combustion) of Solids	B-1 Contained	Radiation	1200°C	Medium High Low	Approx. 1-2 Minutes	Very Low	Fair/Poor (Pre-Set)	Simplicity Reliability Low Cost	Poor Temperature Control Terminal Cooling Difficult/ Hot Briquet
		B-3 Sting B-4 Molten Zone		1200°C (1500) 800°C						
C DIRECT RESISTANCE HEATING	Electrical Resistance of Sample	C-2 Enclosed	Intrinsic	2000°C	Very High	Unlimited	High	Good	High Temps. Controllability Permits Semi-Free Sample	Equipment Requirements Short Time in C-5
		C-4 Molten Zone C-5 Semi-Free		2000°C >2500°C						
D INDUCTION HEATING	Electrical Resistance of Sample	D-1 Contained	Intrinsic	1600°C	Medium High Medium High	Unlimited	Medium	Good	High Temperatures for Extended Time Controllability to Semi-Free and Free Processing	Extensive Equipment Requirements (High Weight) D-1 Non-conductive Container Material D-6 Dev. of Free Processing System
		D-3 Sting		2000°C			Very High			
		D-4 Molten Zone		2000°C			High			
		D-6 Free		Unlimited			Very High			
E ELECTRIC DISCHARGE HEATING	Capacitor Discharge	E-5 Semi-Free	Intrinsic	Unlimited	Extremely High	<1 Sec.				Extensive Equipment, Very Small Samples, Matl. Vaporization

(1) All methods except (E) are also adaptable to low temperature.

(2) Based on upper temperature regime near maximum temperature.

Figure 6-1. Major Characteristics of Heating Methods

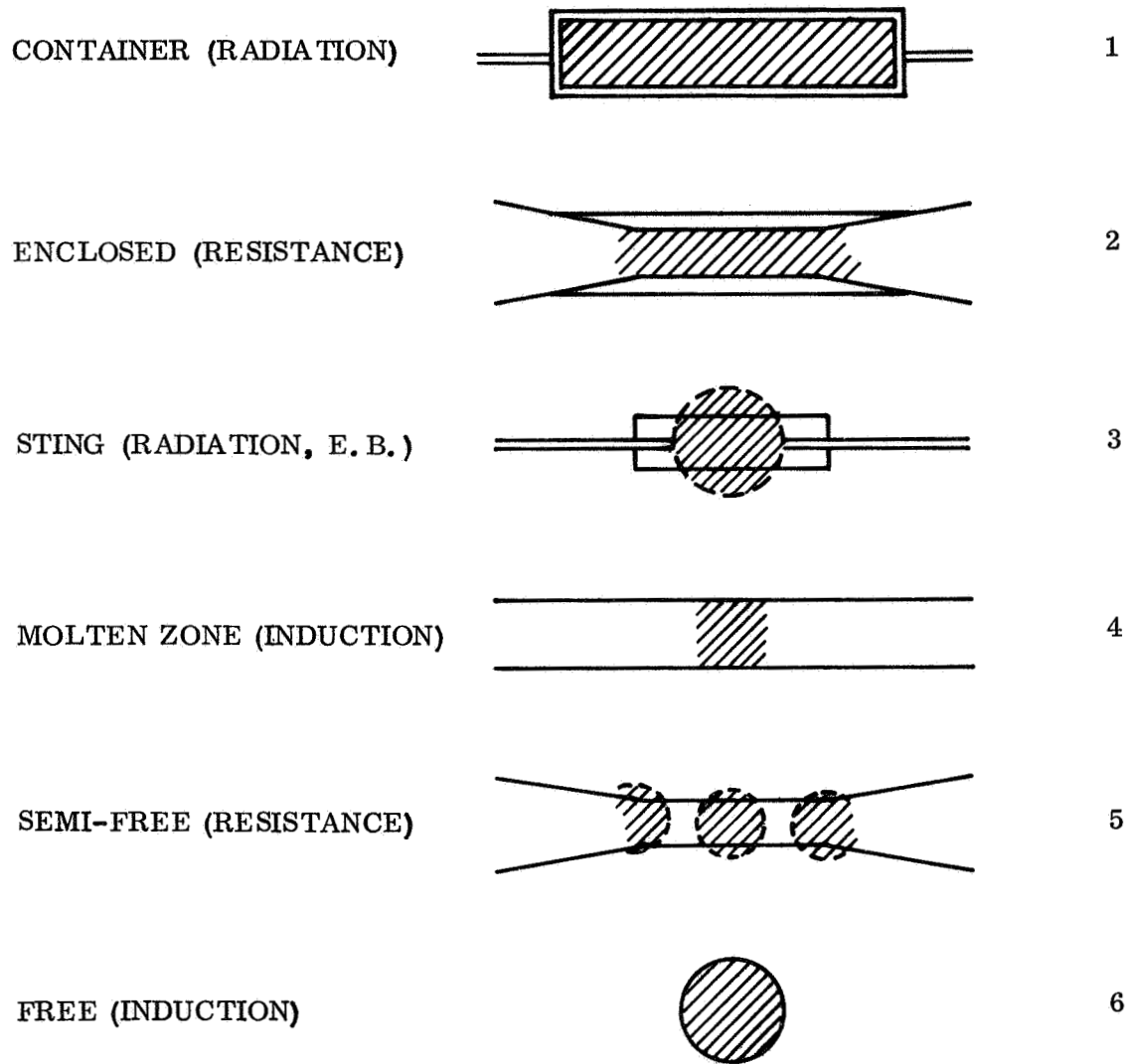


Figure 6-2. Modes of Liquid Material Suspension

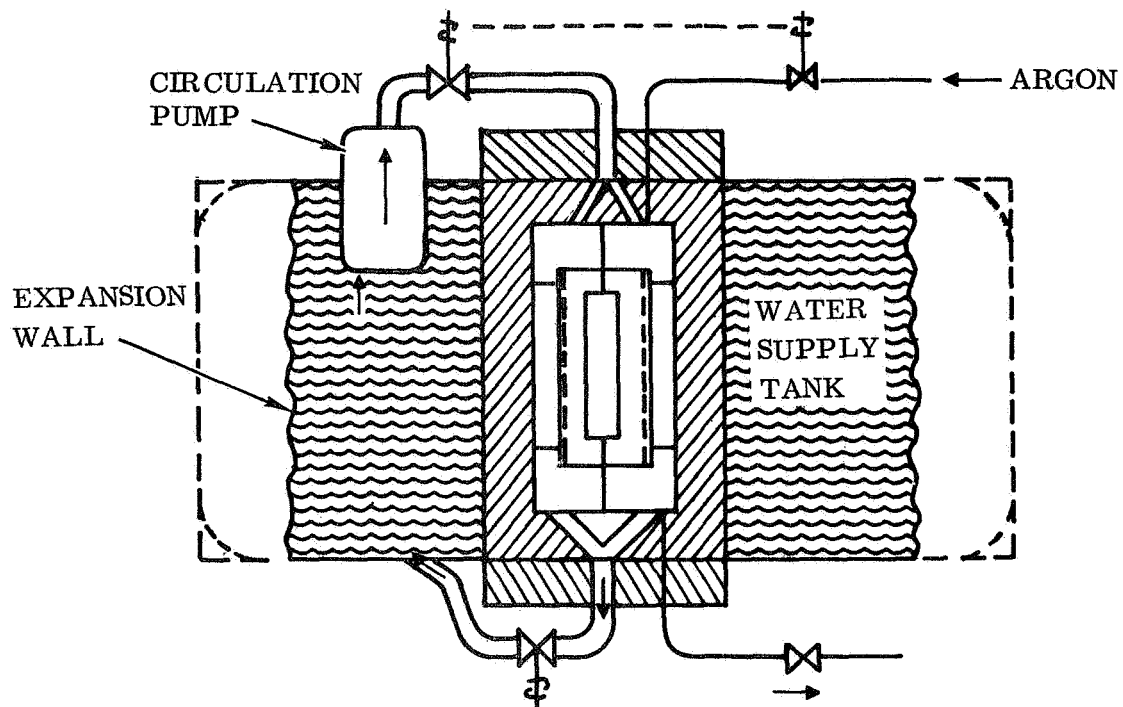


Figure 6-3. Electric Furnace Module EF-1  
with "Closed" Cooling System

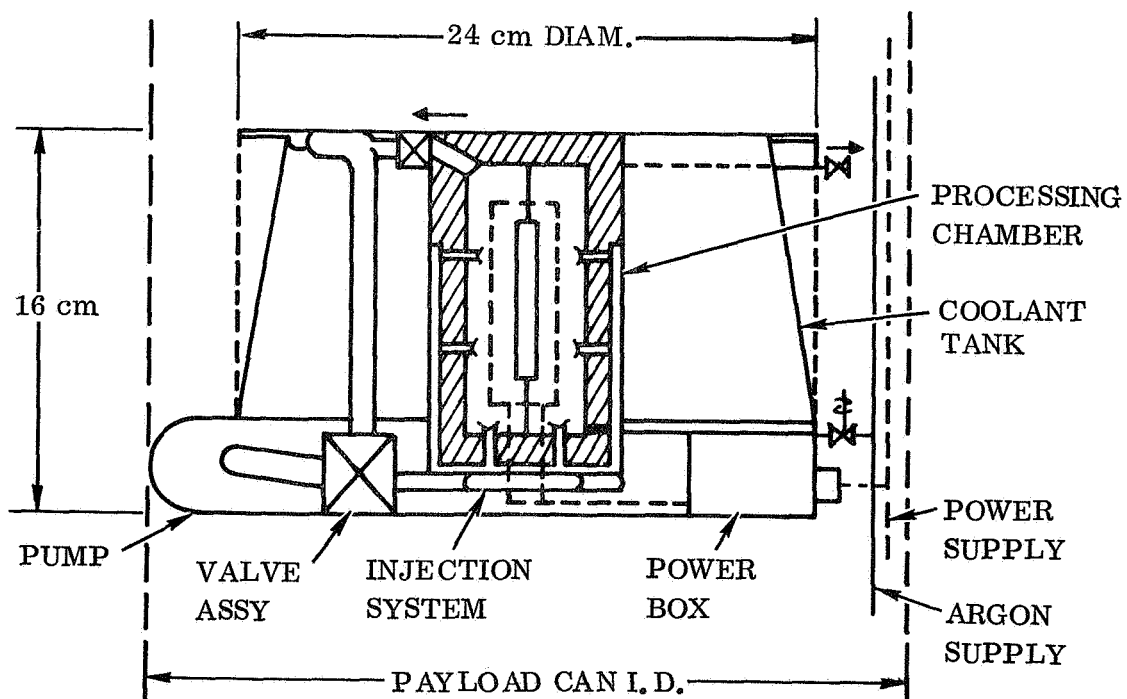
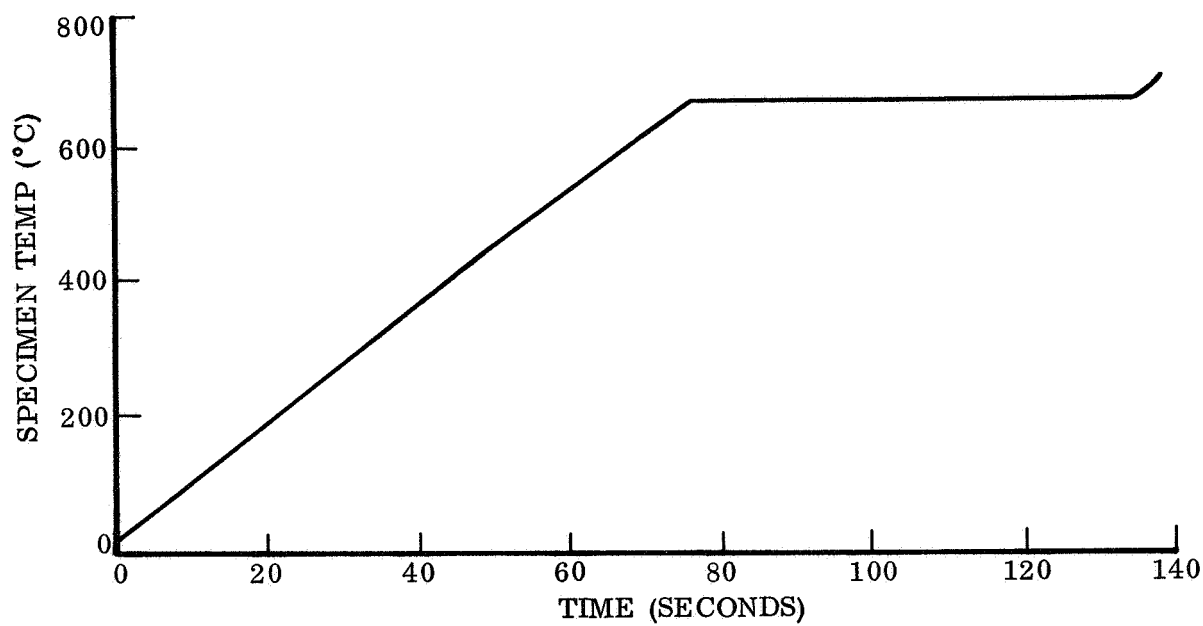
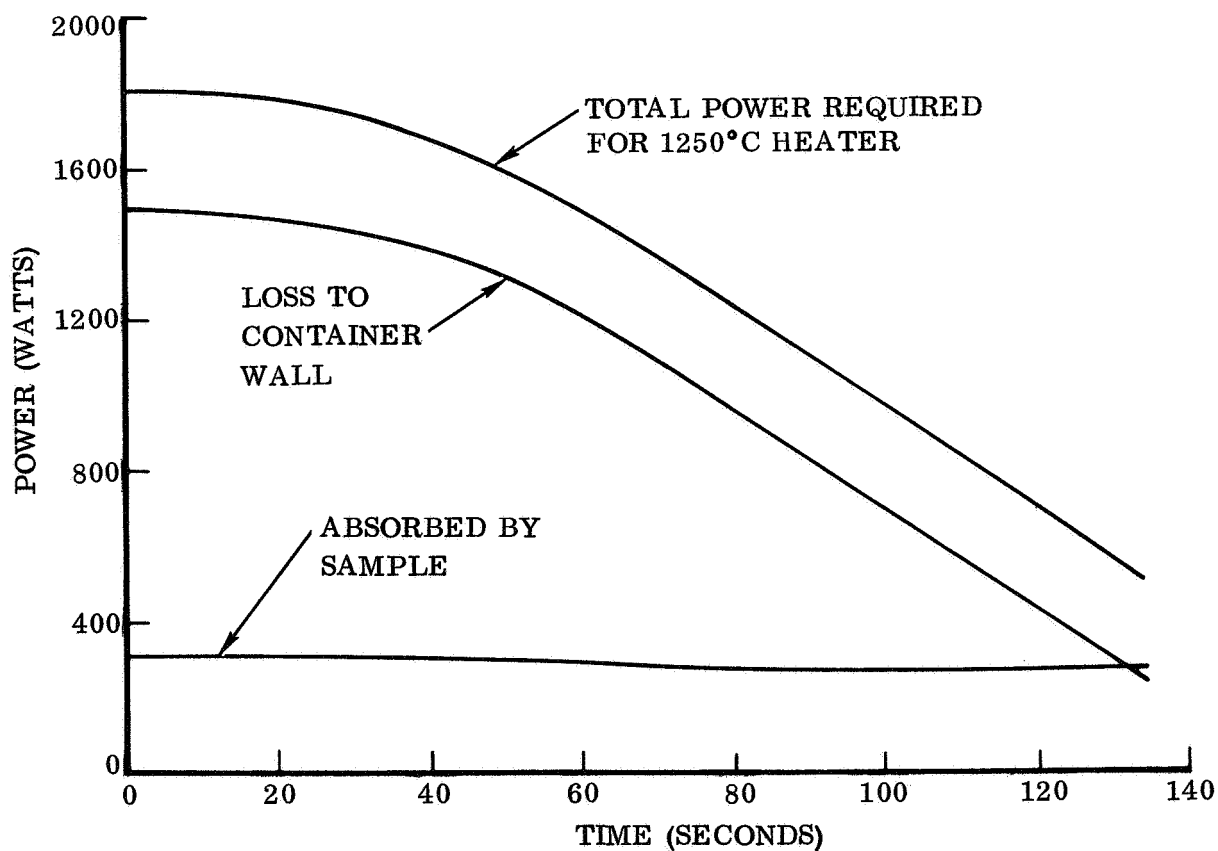


Figure 6-4. Electric Furnace Module EF-2  
with "Closed" Cooling System





(A) SAMPLE TEMPERATURE



(B) ENERGY INPUT (FURNACE)

Figure 6-5. Sample Temperature Profile and Related Energy Input for El. Furnace EF-2 (700°C)

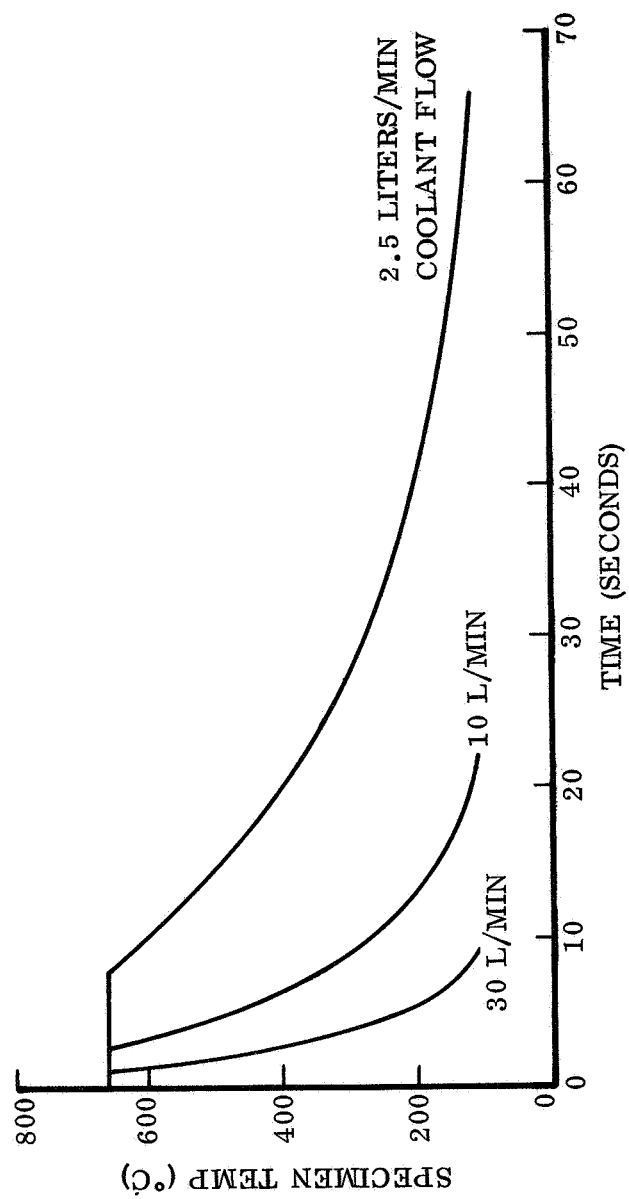
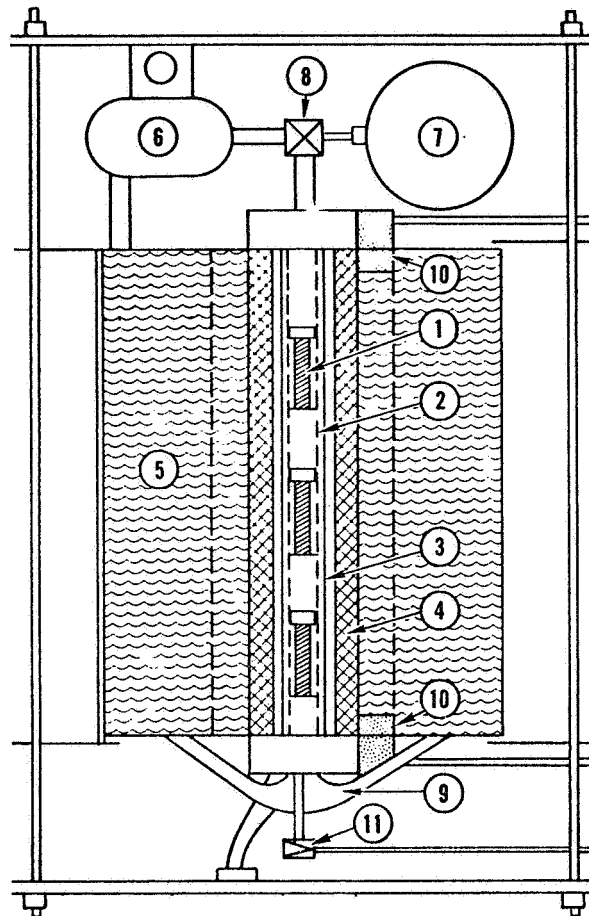


Figure 6-6. Active Cooling Profiles for Aluminum  
Standard (1.2 x 8) Samples



1. EXPANDABLE FOAMING SAMPLES WITH ARRESTING HEADS
2. SAMPLE EXPANSION/ARRESTING GUIDES
3. TUBULAR HEATING ELEMENT (SPLIT)
4. INSULATION
5. WATER SUPPLY
6. WATER CIRCULATION PUMP
7. ARGON SUPPLY
8. 3-WAY VALVE
9. WATER/STEAM RETURN (ROTATES WATER IN 5)
10. STEAM SEPARATOR WITH CHECK VALVE AND VENT
11. ARGON VENT VALVE

Figure 6-7. Tubular Electric Furnace for Multiple Experiments

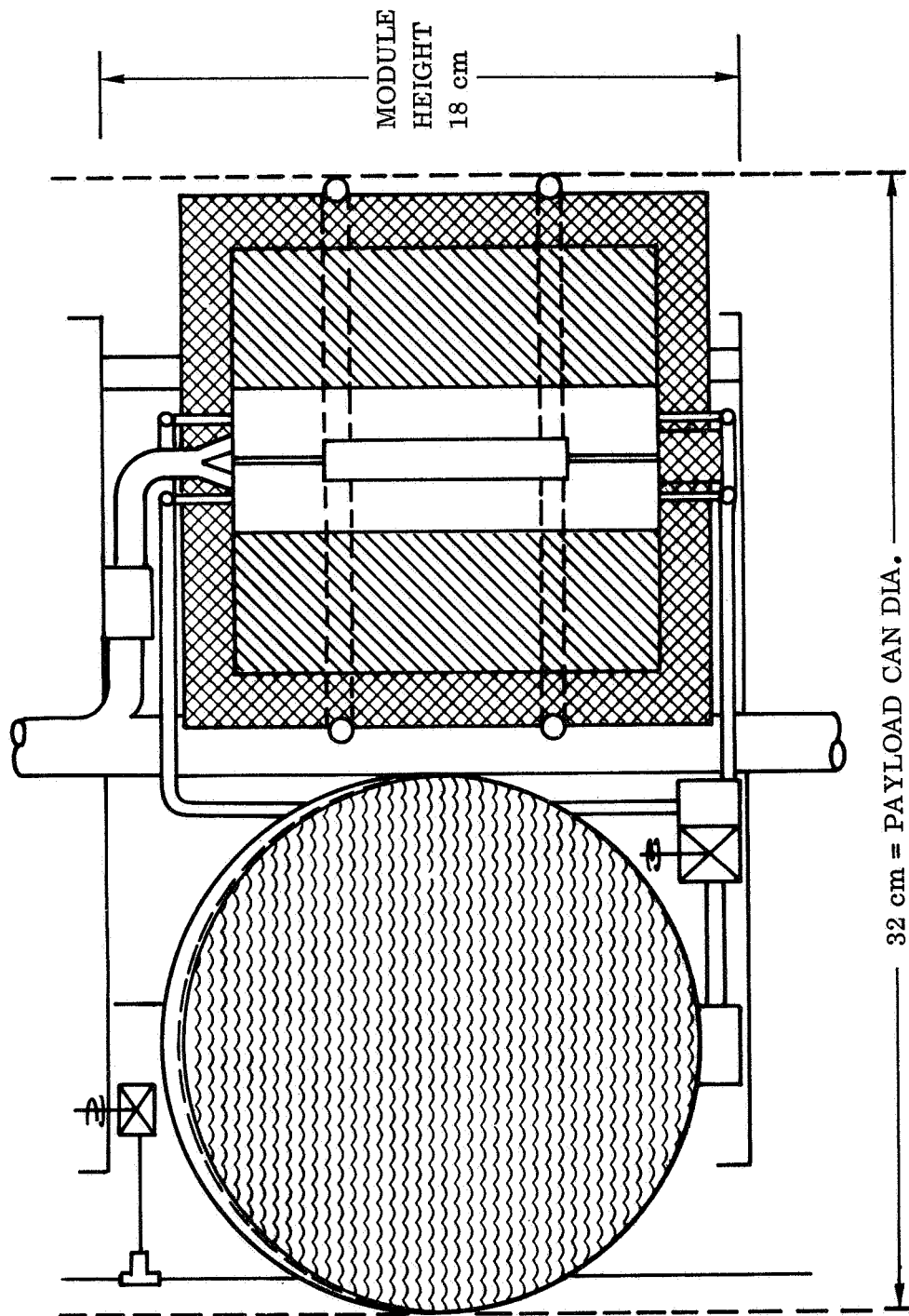


Figure 6-8. Exothermic Furnace Module with "Open" Cooling System

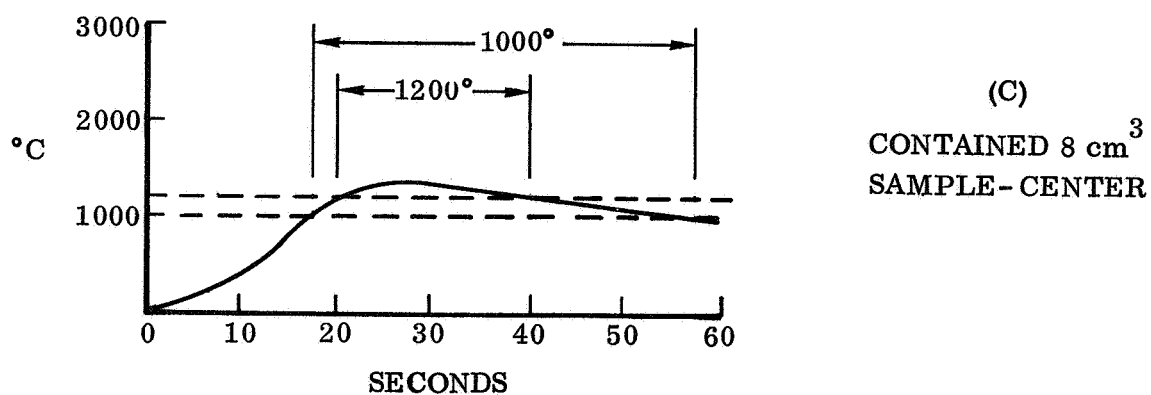
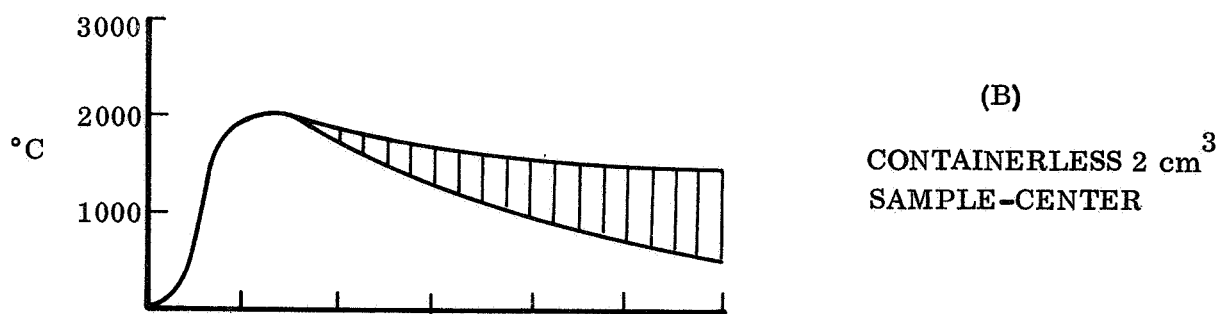
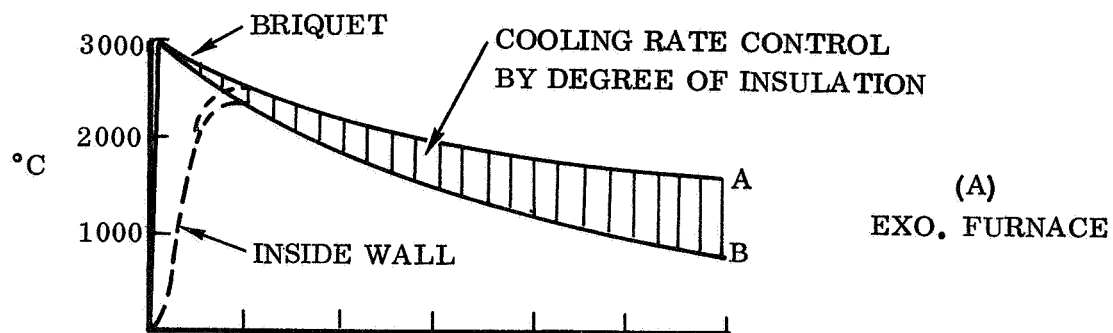


Figure 6-9. Typical Temperature Profiles of an Exothermic Furnace

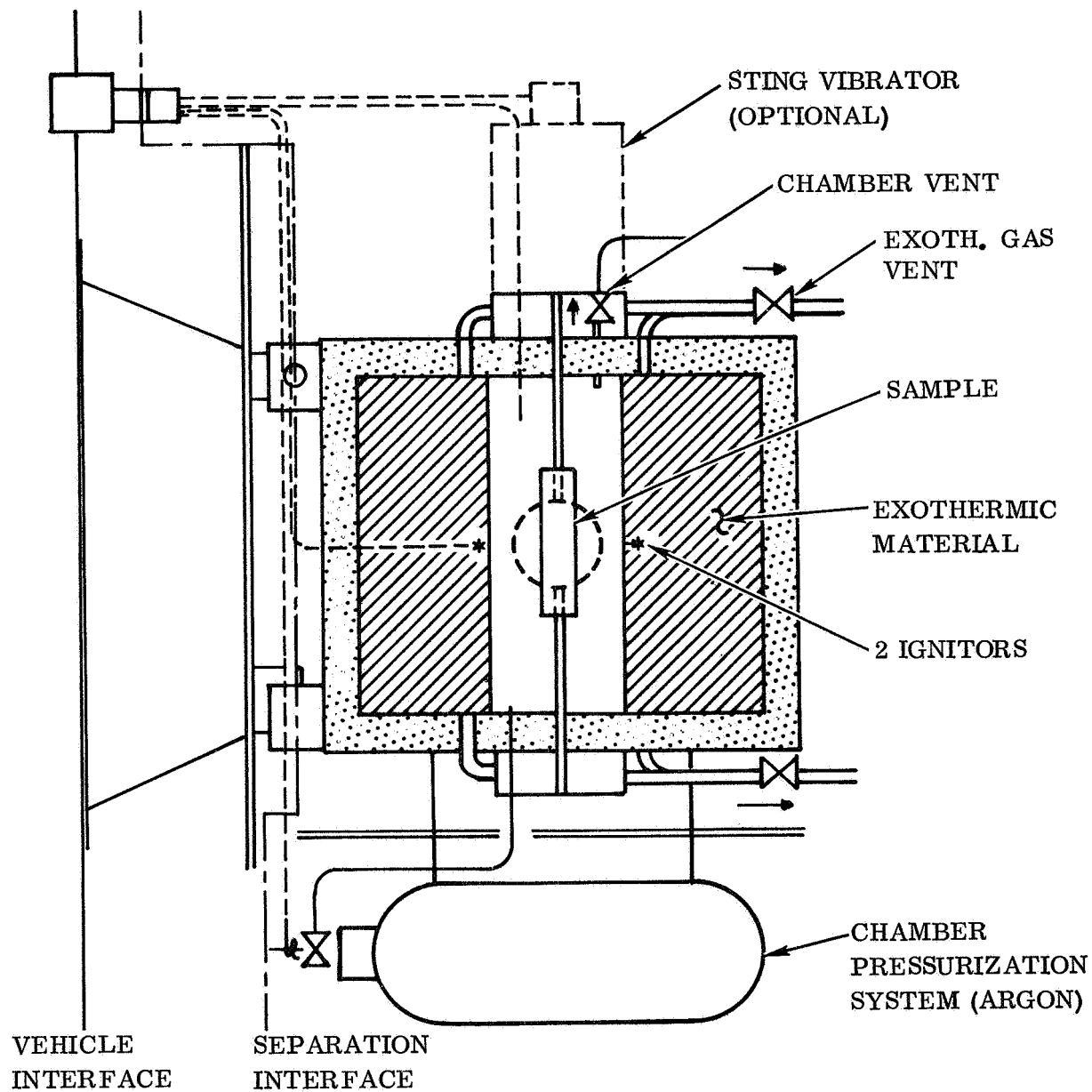


Figure 6-10. Extravehicular Exothermic Apparatus  
for Orbital Experiments

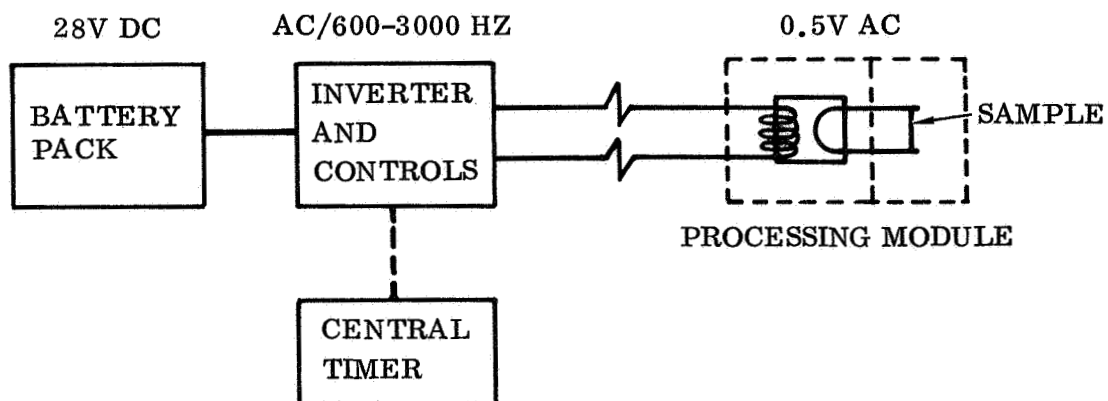


Figure 6-11. Electrical System for Direct Resistance Heating

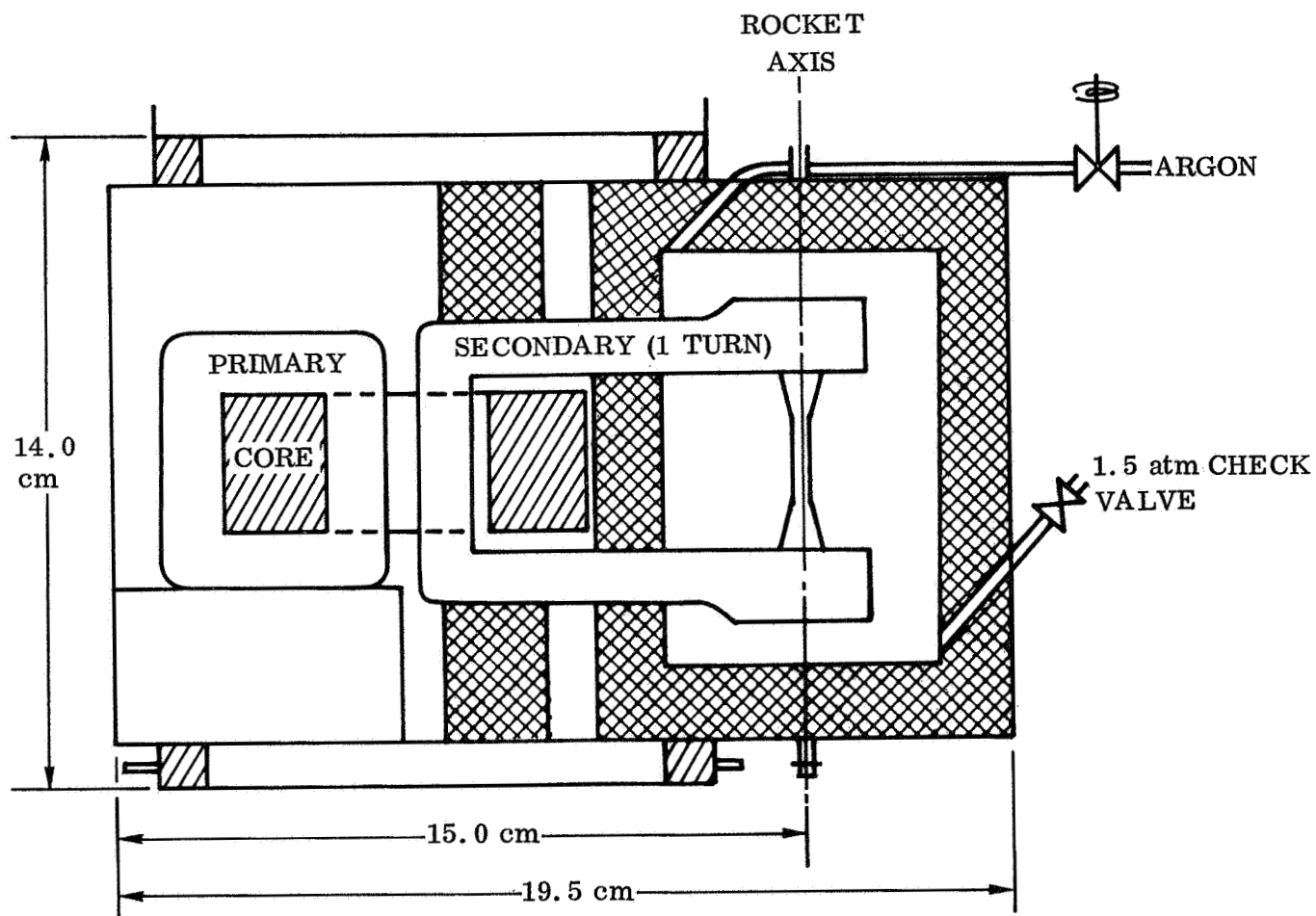


Figure 6-12. Resistance Heating Module ("DR")

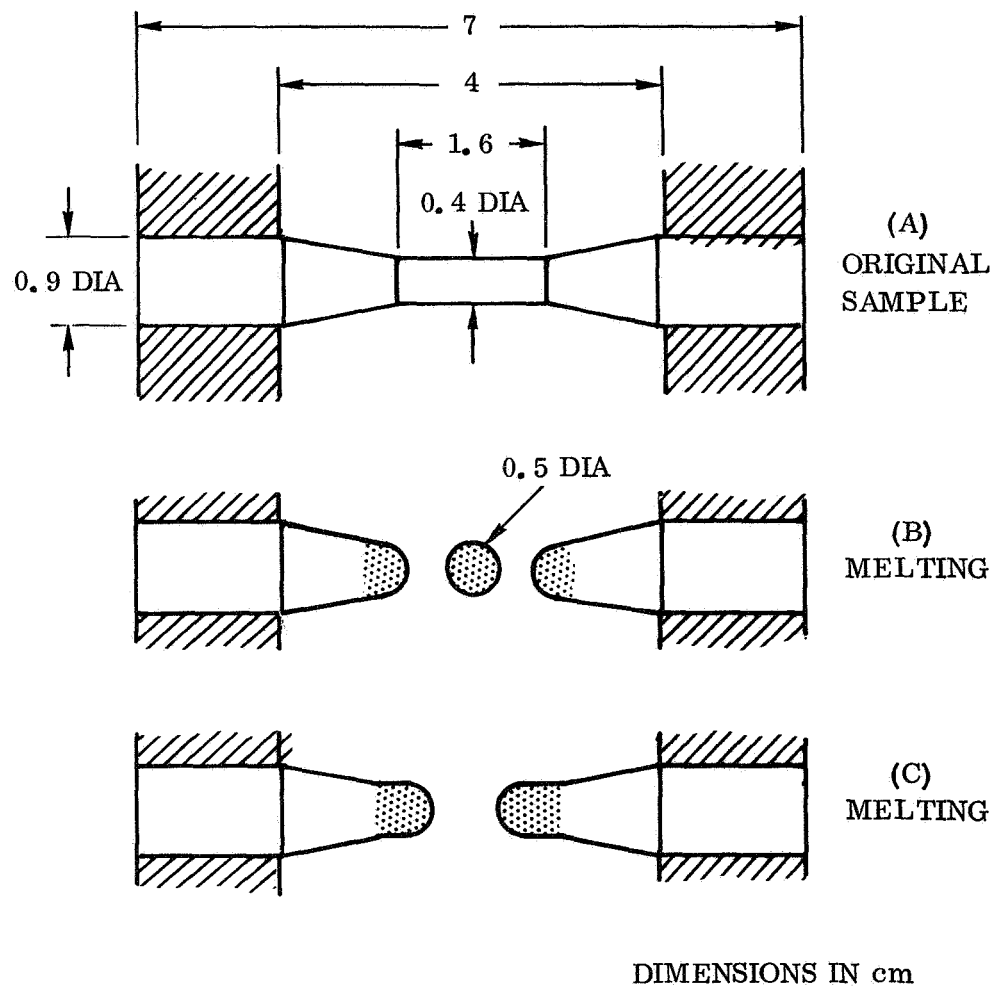
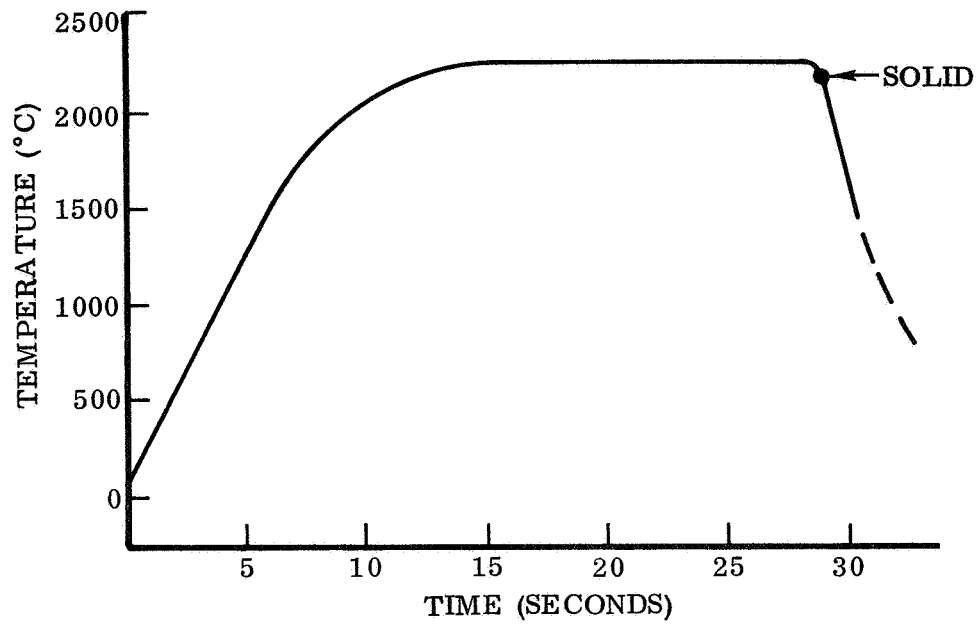
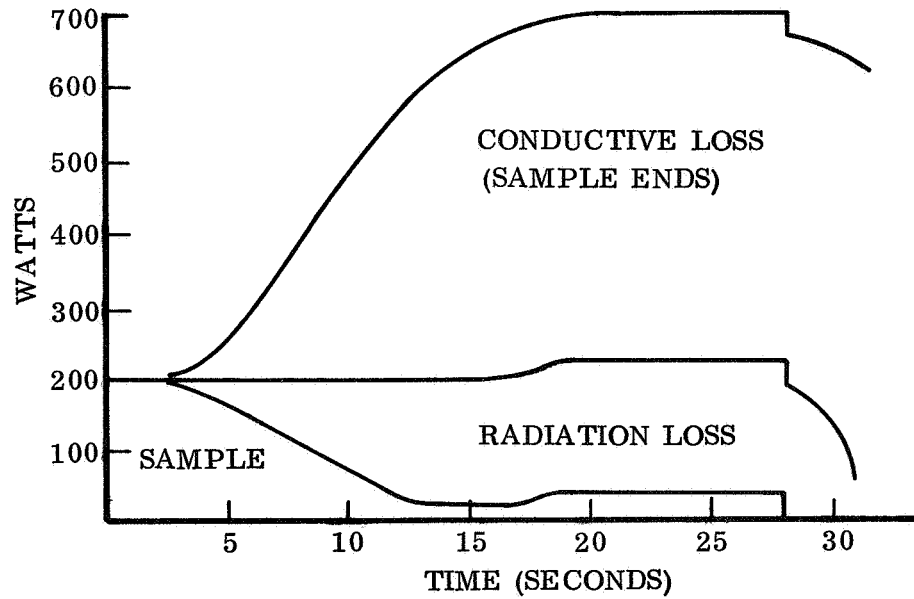


Figure 6-13. Sample Configurations — Direct Resistance Heating





(A) SAMPLE TEMPERATURE



(B) POWER/HEAT

Figure 6-14. Typical Resistance Heating Profile  
0.4 cm. Dia. Sample — 2200°C

Liquid State Temperature	Mode of Sample Suspension	Adaptable To Cooling Rate Control	Passive Solidification	Terminal Cooling Required
Low Melting Temperature (< 600° C)	(1) Container			•
	(2) Enclosed	•	(O)	•
	(3) Sting			•
	(4) Molten Zone	•		•
	(5) Semi-free		(O)	•
	(6) Contact-free		(O)	•
Intermediate Melting Temperature (600–1200° C)	(1) Container			•
	(2) Enclosed	•		•
	(3) Sting		•	(O)
	(4) Molten Zone	•		•
	(5) Semi-free		•	(O)
	(6) Contact-free		•	(O)
High Melting Temperature (> 1200° C)	(2) Enclosed	•	•	
	(3) Sting		•	
	(4) Molten Zone	•	•	
	(5) Semi-free		•	
	(6) Contact-free		•	

Figure 6-15. Cooling Methods Applicable to Various Heating Conditions

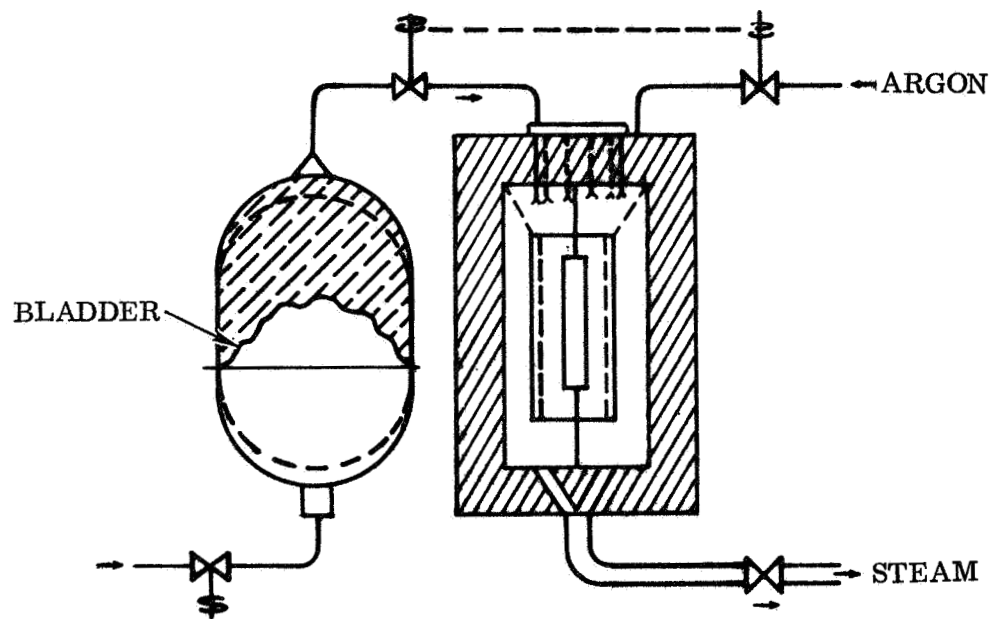


Figure 6-16. "Open" Cooling System

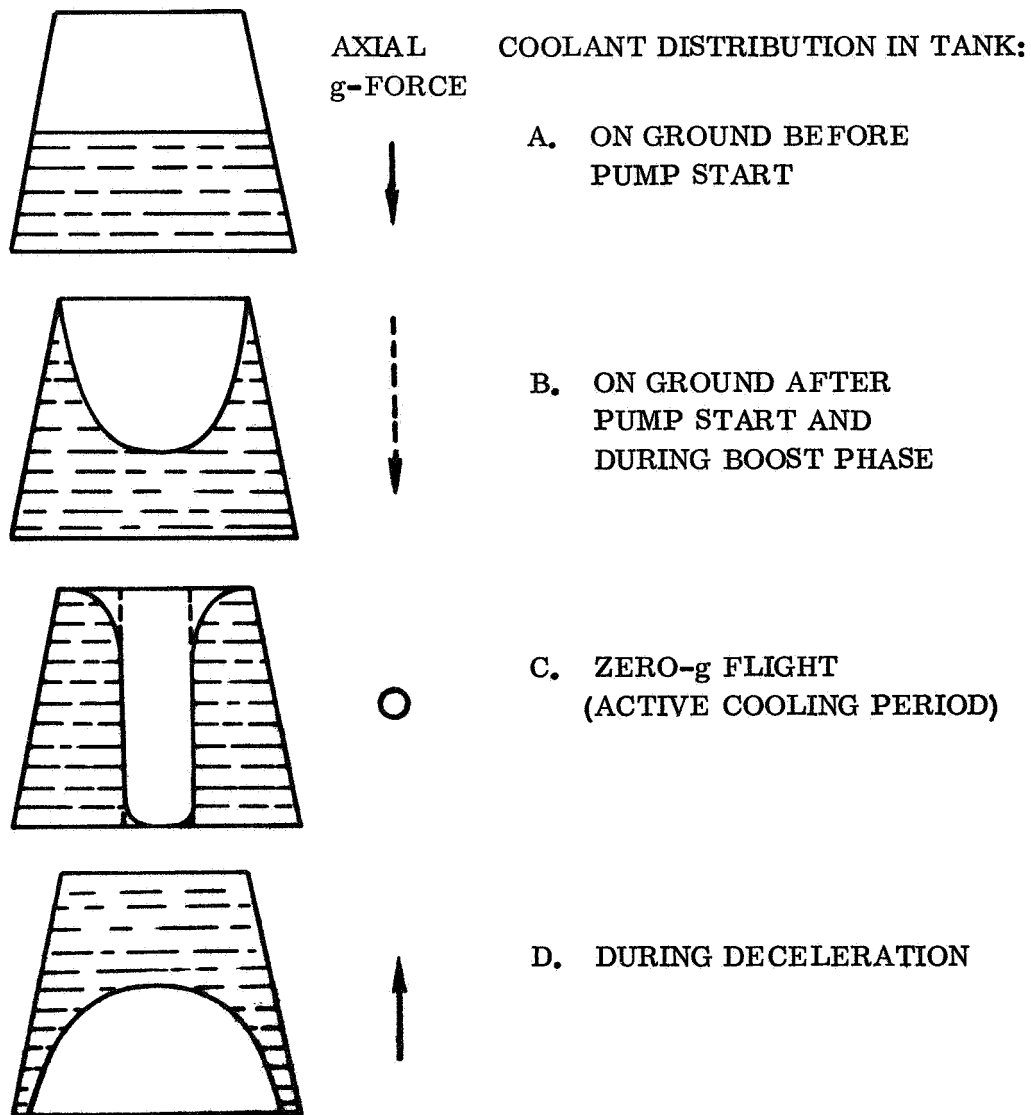
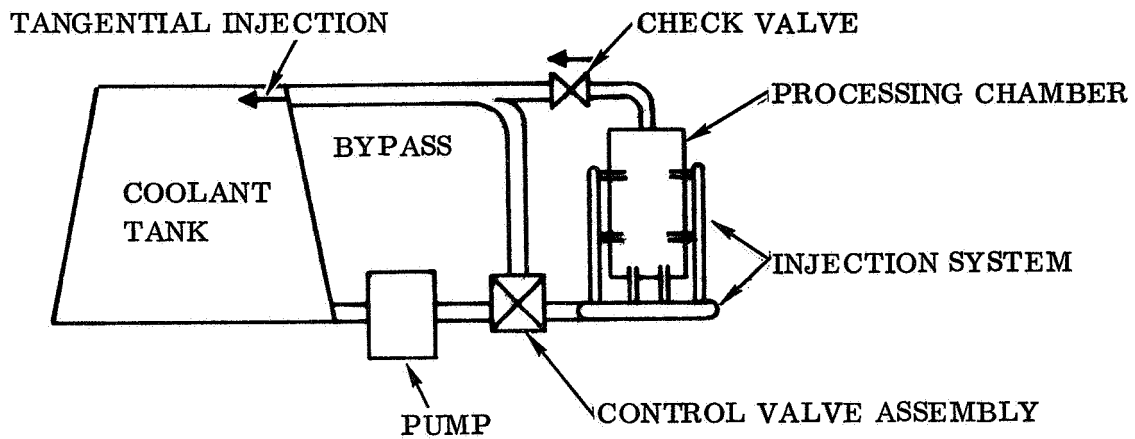


Figure 6-17. Closed Cooling System

## 7.1 ELECTROPHORETIC SEPARATION OF BIOCHEMICALS - STATIONARY

### 7.1.1 Process Definition and Objectives

The separation of serum proteins by electrophoretic methods is a powerful tool for medical research and clinical analysis. Electrophoresis can also be used for preparative purposes to obtain enzymes, viruses, vaccines, isotopes and similar materials of biological, chemical and physical interest.

There are problems and disadvantages of the technique, however. Among these are stabilization of the boundaries of the migrating particles, boundary anomalies, and incomplete separations. If inert support materials are used, separation and identification problems may be reduced, but at the expense of isolating the desired materials. The product may adhere too strongly to the separating medium, or, if it moves more freely, the effects of gravity may cause material to settle out on the bottom of the apparatus and interrupt the experiment or otherwise complicate the interpretation. Also the passage of electric current through the ohmic resistance of the conducting medium results in the formation of thermal gradients. Under gravity conditions, this results in convection currents and causes the separated components to remix.

Separation of small analytical amounts of material is sometimes easy to achieve. However, the isolation of larger quantities of pure product is normally more difficult. When dealing with biologically active materials such as enzymes, care must be taken to prepare pure products without altering their biological properties. This is especially important when preparing materials for human use.

Many advantages should accrue by processing materials in space. Convection caused by thermal gradients should become unimportant, and sedimentation effects of gravity will disappear. This will result in higher resolution and allow greater throughput in shorter time. The stability of sensitive and easily degradable biological materials will thereby be improved. Preliminary experiments with human kidney cells have shown that products will be obtained which cannot be obtained under gravity conditions.

It is therefore of prime interest to study electrophoretic processes in space where the effects of gravity are minimal. The objectives of the experiment are:

1. To perform separations of biological materials at zero gravity by methods known to be subject to the effects of gravity.
2. To examine the separations of proteins by analytical and semi-preparative electrophoretic techniques under low-g and compare results with those obtained under one-g.
3. To demonstrate the change in resolution and the possibility for large scale preparations when separations are performed in low-g. This will be done by performing the separation in a range of column diameters.
4. To evaluate the results of low-g and one-g electrophoresis with respect to speed, resolution, purity, stability, amount and effect of temperature and gravity level.

#### 7.1.2 Verification Requirements

The verification required for these experiments is to determine whether the separated products are more concentrated or more sharply resolved under zero-g processing than under terrestrial processing. For applicable materials the rate of travel through the column and the stability (activity) of the product must be established.

To determine the extent to which the objectives have been obtained, the following specific verification requirements must be met:

1. Processing Requirements
  - (a) Voltage level to optimize separation rate
  - (b) pH of buffer to optimize mobility
  - (c) Low temperature to suppress diffusive mixing
2. Verification of material properties
  - (a) Demonstrate chromogenic separation
  - (b) Determine concentration of chromogens
  - (c) Distance of chromogen travel

- (d) Rate of chromogen travel
- (e) Sharpness of chromogen bands
- (f) Amount of material separated
- (g) Potency of product
- (h) Ultraviolet absorptivity of products

During the prelaunch period the optimum level for the operating parameters will have been determined. At check-out time prior to launch, the experiment will be set up to function at the predetermined levels so that continuous monitoring will not be required. The recording of the actual voltage, amperage, temperature, and ultraviolet absorption during flight will be a convenience, but not necessary for the success of the experiment. When large separations are performed control of the operating parameters during the course of the experiment may become important. Therefore requirements for two verification levels may be distinguished.

#### Verification Level I.

Demonstration of the separation of chromogenic substrates on a series of columns of different diameters, and evaluation of resolution purity, speed and buffer and temperature effects.

#### Verification Level II:

Actual separation of preparative quantities of mixtures of practical materials on the demonstration size or larger columns.

#### 7.1.3 Experiment Materials

The materials selected for initial separations will be well characterized substances with known properties. The following criteria will be the basis for their selection:

1. Each component must have a distinctive property such as color or absorption spectrum by which it may be easily identified.
2. The electrophoretic mobility of the components must differ widely from each other.
3. Buffers must be non-gassing.

4. The samples must remain stable during the prelaunch period including storage, set-up and checkout time.

Color is a very characteristic property and is easily registered on photographic film. Separations of colored materials are thus readily documented and simply analyzed. Beyond the visible region, ultra-violet absorption of many materials provide a convenient analytical tool. Time-dependent data can be obtained from a simple UV source and photocell pick-up, or gross overall data may be obtained in the laboratory.

Chromogenic components which form visible bands and are simple, stable easily identified substances are:

Bromphenol blue

Hemoglobin

Albumin

Mobilities of the above materials vary greatly in the following way.

Bromphenol blue has the smallest molecular weight. It is therefore the most mobile.

Albumin is a large molecule and rather slow; it can be stopped by a membrane.

Hemoglobin is the slowest moving component in an electric field; it will be found closest to origin and perhaps remain on the column.

#### 7.1.4 Material Quantities, Sample Size, Configuration

These characteristics must be chosen so that the evaluations can be made within the time limits required for the flight. The flight vehicle dimensions determine the allowable amount of material and size of apparatus which can be used to accomplish the objectives in the allotted time. The flight time will determine whether a simple separation will be obtained, with the sample material remaining on the column, or whether isolation of substances is achieved. Isolation can be accomplished by either completing the electrophoresis and discharging the product into a container at the end of the column, or the column support material can be ejected and the colored bands sliced into discs. The active material in the discs can then be extracted by solvents.



The tubes comprising the column will be 12 cm long and will vary in diameter as follows:

0.5 cm

1.0 cm

2.0 cm

4.0 cm

The sample holder consisting of a thin plastic sliding plate will contain holes matching the I.D. of the columns. The thickness of the plate will be 2 mm. The samples will be stored in the holes in the plate which will be sandwiched between a tube holder plate and a plate comprising an end of the buffer container. The volume of the starting material for the different column will therefore be:

Column D (cm)	0.5	1.0	2.0	4.0
Sample V (cm <sup>3</sup> )	0.04	0.16	0.63	2.51
Column L (cm)	12	12	12	12
Column V (cm <sup>3</sup> )	2.36	9.42	37.7	1.51
Receiver V (cm <sup>3</sup> )	0.10	0.40	1.60	6.40
Buffer V (cm <sup>3</sup> )	3000	3000	3000	3000
Coolant V (cm <sup>3</sup> )	3500	3500	3500	3500

where D, V, and L represent diameter, volume and length respectively.

The buffer volume is chosen large enough that essentially no change occurs in concentration or pH during the course of the experiment. It is a non-gassing buffer but the amount and configuration is chosen so that any gas which might be generated is trapped and easily removed. The inlet buffer reservoir and the outlet buffer reservoir are each common for all the columns.

Cooling water is recycled by means of a pump. Initially some of the water will be present as ice. The heat of fusion will supply the refrigeration for small samples and short experiments. Large scale preparations and continuous separations will require mechanical equipment with a greater continuous refrigeration capacity.

### 7.1.5 Experimental Process Phases

The experimental process phases for the electrophoretic separation are simple and straightforward. Many of the phases may be done before or after the flight has been accomplished. The essential operations, which do not differ for any practical process are as follows:

1. Prepare materials - substrate, buffer, coolant, film
2. Equilibrate temperature
3. Load apparatus
4. Insert samples
5. Apply power
6. Record data
7. Isolate samples
8. Unload materials
9. Evaluate results

The timewise sequencing of individual process phases and events is illustrated or defined in the preliminary time diagram Fig. 7.1-1. The series of steps arrange themselves naturally into four (4) groups. This arrangement is shown graphically in the Process Phase Flow Diagram Fig. 7.1-2.

7.1.5.1 Laboratory Preparations. The experiment apparatus and materials can be easily assembled well ahead of time. The assembly operations consist of:

1. Prepare buffered gel-filled column
2. Prepare substrate mixture and stabilize
3. Assemble columns into apparatus
4. Check out electrical and mechanical operations

At the conclusion of this period a delay of up to two weeks can be tolerated.

7.1.5.2 Ground Operations. Ground operations consist of loading the apparatus into the flight vehicle.

1. Load apparatus into flight vehicle
2. Install camera
3. Add coolant and equilibrate temperature
4. Check apparatus and camera

After installing the apparatus and camera or other recording equipment the samples are removed from storage and placed into the sample holders. Then refrigerated coolant is cycled through the apparatus until a uniform low temperature prevails around the columns and sample. This temperature is maintained for the duration of the flight.

7.1.5.3 Low-g Operations Consist of

1. Insert samples into columns
2. Apply power
3. Remove samples

If adequate time is available in a flight the samples will traverse the column and collect in receivers. Short time experiments require that the bands of separated material be photographed with respect to location and distinctness of separation. The column filling can also be ejected and analyzed zone-wise if necessary.

7.1.5.4 Post-Test Operations. Consist of recovery of the samples and evaluation of the extent of separation, sharpness of bands, speed of separation as measured by distance travelled and sharpness of the bands on the film.

7.1.6 Low-g Test Requirements

7.1.6.1 Low-g Time Requirements. The time required to achieve useful results from a low-g electrophoretic separation is quite different for the two planned verification levels. The velocity of simple ions in water solution is in the range  $1 \times 10^{-3}$  cm per second under a potential gradient of one volt per centimeter. If an electric field of 1200 volts is applied to a 12 cm column the potential gradient will be roughly 100 volts per centimeter. The distance travelled by a simple substrate will be theoretically one millimeter per second.

Larger particles such as cells and proteins move more slowly. Water is attached to most particles in solution; they are hydrated and move slower because they must drag along water of hydration as they move. The motion is further complicated by the presence of multiple charges, polarization effects, temperature, and viscosity effects which may serve to increase or retard the motion. In general, we may assume a distance travelled by the electrophoresis substrate to be less than 0.1 mm per second.

The time required for the separation and isolation of substantial quantities of substrate material is large compared to the time available in present low-g research facilities. However the requirements are most modest for demonstrating the difference in the course of the phenomena when performed in a one-g and a low-g environment.

A separation of about half millimeter or less is sufficient to allow measurements to be made. If we assume that a potential difference of 120 volts per 10 cm, which is not uncommon, and conservatively estimate a speed of  $5 \times 10^{-5}$  mm/volt-second, then an evaluation can be made at verification level I in 360 seconds. A lower voltage will result in a separation which is too slow and a higher voltage leads to problems with heating and gassing. The heat enhances diffusion, and leads to rapid deterioration in the quality of results obtained.

For verification level II, the chief difference in the processing profiles will be the period during which zero-g is effective; the size of the columns may be changed or selected according to results achieved at verification level one. Larger samples can be used in wider diameter columns. However, as the columns diameter increases so does

the non-uniformity of column support materials. This non-uniformity applies to the packing concentration and density as well as invisible flow channeling and diffusion behavior which blurs the line of demarcation for the zonal fronts. This effect which should be less evident in zero-g will be accentuated by the use of wide columns and maximum low-g periods for verification level II. The time required to achieve an actual separation at level II and capture effluent in the receiver requires tens of minutes. However, because the substrates separate into bands which move at various speeds, the experiment may be interrupted at any time before the product reaches the receiver. The recovery of the sample and analyses can then still generally be made by ejecting the column support and cutting it into discs. These discs can then be separately evaluated.

The time requirements may therefore be set out as follows to accomplish the objectives:

Verification level I	360 seconds
Verification	Substrate color separation and identification
Verification level II	Ila. 360 sec, I Ib: 1200 sec
Verification	substrate separation and isolation

The power requirements are at the same level for each experiment but the experiment lasts longer at level II as follows:

Level I	1200 volts, 0.200 amps	240 watts
	240 watts, 360 sec	24 watt-hour
Level II	1200 volts, 0.200 amps	240 watts
	240 watts, 1200 sec	80 watt hour

7.1.6.2 Required g-Level. The g-level which is necessary for the performance of electrophoresis has no strict cut off level. All material is contained in apparatus and is confined. Success therefore depends on attaining g-levels sufficiently low that the effects may be detected and evaluated from results at one-g. On the basis of past experience and expected behavior the following maximum g-levels have been defined.

Verification Level I: A g-level of  $1 \times 10^{-4}$  maintained during the power-on period is desired.

Verification Level II: A g-level of  $1 \times 10^{-5}$  during the power-on period is desired but the level may vary to as low as  $1 \times 10^{-3}$  g with increased difficulty in evaluation.

#### 7.1.7 Low-g Test Facilities and Experiments

7.1.7.1 Low-g Time Requirements. A comparison of the time required with the low-g test facilities available show that Level I experiments can be carried out in a research rocket class 4 and the maximum WSMR Trajectory B. The use of several columns in a single apparatus and the different sizing of each column gives the effect of repeated and multiple experiments with the added advantage that conditions are positively the same with respect to temperature, concentration and g-level for each experiment (column). A comparison of the weight, size and zero-g time in the table shows that two apparatus assemblies (8 individual experiments) can be accommodated without difficulty. The zero-g time for the Level II experiments is too short however so that column ejection with disc slicing will be required (IIa) because time does not allow the complete traverse of the column by each component in turn. To achieve this level will require a suborbital or Skylab type facility.

<u>Levels I, IIa</u>	<u>Low-g Time</u> (sec)	<u>Weight</u> (kg)	<u>Height</u> (cm)	<u>Energy</u> (Wh)
RR-4-B Capability	390	125	150	110
2 Experiment Modules	360	48	60	63
Support Module		52.5	70	10
Total	360	100.5	130	73
Contingency	30	24.5	20	37

A weight difference for the Level II experiments owing to increased cooling requirements for possible longer periods or greater power because of higher possible concentrations may occur.

7.1.7.2 Definition of Facilities and Experiments. The two types of experiments will be carried out in a single facility until greater capabilities become available. This will be as follows:

Low-g Facility -	RR-4 Trajectory B
Number of Experiments -	Eight (8) experiments, simultaneously
Required Low-g Time -	360 sec

#### 7.1.8 Apparatus and Payload Definition

7.1.8.1 The Basic Processing Apparatus shown in Fig. 7.1-3 consists of four (4) rigid plastic tubes of 0.5, 1.0, 2.0, and 4.0 cm diameter. These tubes are supported in thick plastic end plates at top and bottom. A thin plastic slide closes the ends of the tubes. These slides are supported by frame plates which match the end-plates on the tubes. Ring seals prevent leakage between the plates and the movable slide. A plastic reservoir attached to the frame plates hold a buffer reservoir of low-concentration polyacrylamide gel. Each reservoir contains a platinum electrode enclosed in a receptacle for handling any gas. Reinforcing bars are used to impart rigidity to the apparatus and provide dimensional stability.

The slides are attached to pneumatic pistons which, when actuated push the samples into alignment with the column. The processing module is thus composed of:

1. Separation columns
2. Buffer reservoirs
3. Electrodes
4. Coolant system

The size and weight of this system together with the power requirements are:

Power input (W)	240
Dimensional envelope (cm)	20 x 15 x 25
Approx. weight (Kg)	7

7.1.8.2 Apparatus Assembly. The complete apparatus assembly consists of the basic processing apparatus (7.2.8.1), a pneumatic actuator system, a camera and camera lights. The total weight of the apparatus assembly is as follows:

Processing Assembly	8 kg
Pneumatic Actuators	2
Gas Supply System	5
Camera	6
Camera Lights	1
Support Structures	2
	<hr/>
Total	24 kg
Axial Height	35 cm

7.1.8.3 Support Module. The outfitting and weight of the support module for Level I and IIa experiments are as follows (numbers in parenthesis identify components specified in Section 5.2.2 and Table 5-1):

Basic Structure (1, 2, 3)	33.0 kg
1 Battery Pack (4)	3.0
Power Conditioning	10.5
(5A, B, C-1, D)	
Programmer, Recorder (6, 7)	4.0
Contingency (8)	2.0
	<hr/>
Total Weight	52.5 kg
Total Axial Height	70 cm
Net Space Available for Apparatus Assembly (Axial Height)	80 cm

7.1.8.4 Payload Assembly. The complete payload is illustrated in Fig. 7.1-4. The major payload data are:



### Payload Weight

2 Apparatus Assemblies	48	kg
Support Module	52.5	kg
Total*	100.5	kg
RR-4 Trajectory B Capacity	125	kg
Contingency	24.5	kg

### Payload Space (in Axial Height)

2 Apparatus Assemblies	70	cm
Support Module	70	cm
Total	140	cm
RR-4 Capacity	150	cm
Contingency	10	cm

### Power Requirements

No. of Apparatus Assemblies	1	2
Maximum Discharge Rate (W)	340	630
Total Consumption (Wh)		
Level I (360 sec)	45	73
Level II (600 sec)	67	115

\*For Level I experiments with two apparatus assemblies, two battery packs are required which increases the payload weight by 3 kg to 103.5 kg.

### 7.1.9 Experiment Performance

7.1.9.1 Pre-test (Ground) Operations. The experiment can be prepared in the laboratory as described in Sections 7.1.5.2 and 7.1.6.1. The following operations remain at the launch site:

- (11) Load samples
- (12) Circulate refrigerant

- (13) Check power and circuits
- (14) Load, install and check camera and lights

7.1.9.2 Test (Flight) Operations. At rocket cut-off, the following operations are programmed.

- (21) Actuate camera
- (22) Actuate piston and slide
- (23) Energize electrical power to columns
- (24) Retract slide
- (25) Cut off electrolysis current
- (26) Stop camera

7.1.9.3 Post-Test (Ground) Operations

- (31) Remove samples
- (32) Recover film

7.1.9.4 Time Diagrams . The sequence of experiment phases and events is shown in the detailed time diagram Fig. 7.1-5.





TOTAL FLIGHT TIME	
LOW-G TIME	
PRELAUNCH	CHECK ALL CONNECTIONS LOAD SAMPLES AND SUPPORT MATERIALS WITHIN THE TUBES AND SLIDES
EQUILIBRATE TEMPERATURE	
INSERT SAMPLES	Δ
ENERGIZE POWER	
UV OBSERVATION	
PHOTO OBSERVATION	Δ Δ Δ Δ Δ Δ Δ Δ Δ Δ
REMOVE SAMPLES	Δ
DEENERGIZE CIRCUIT	Δ
POWER OFF	Δ
POST LAUNCH	RECOVER SAMPLES AND STORE FOR ANALYSIS

Figure 7.1-1. Preliminary Time Diagram

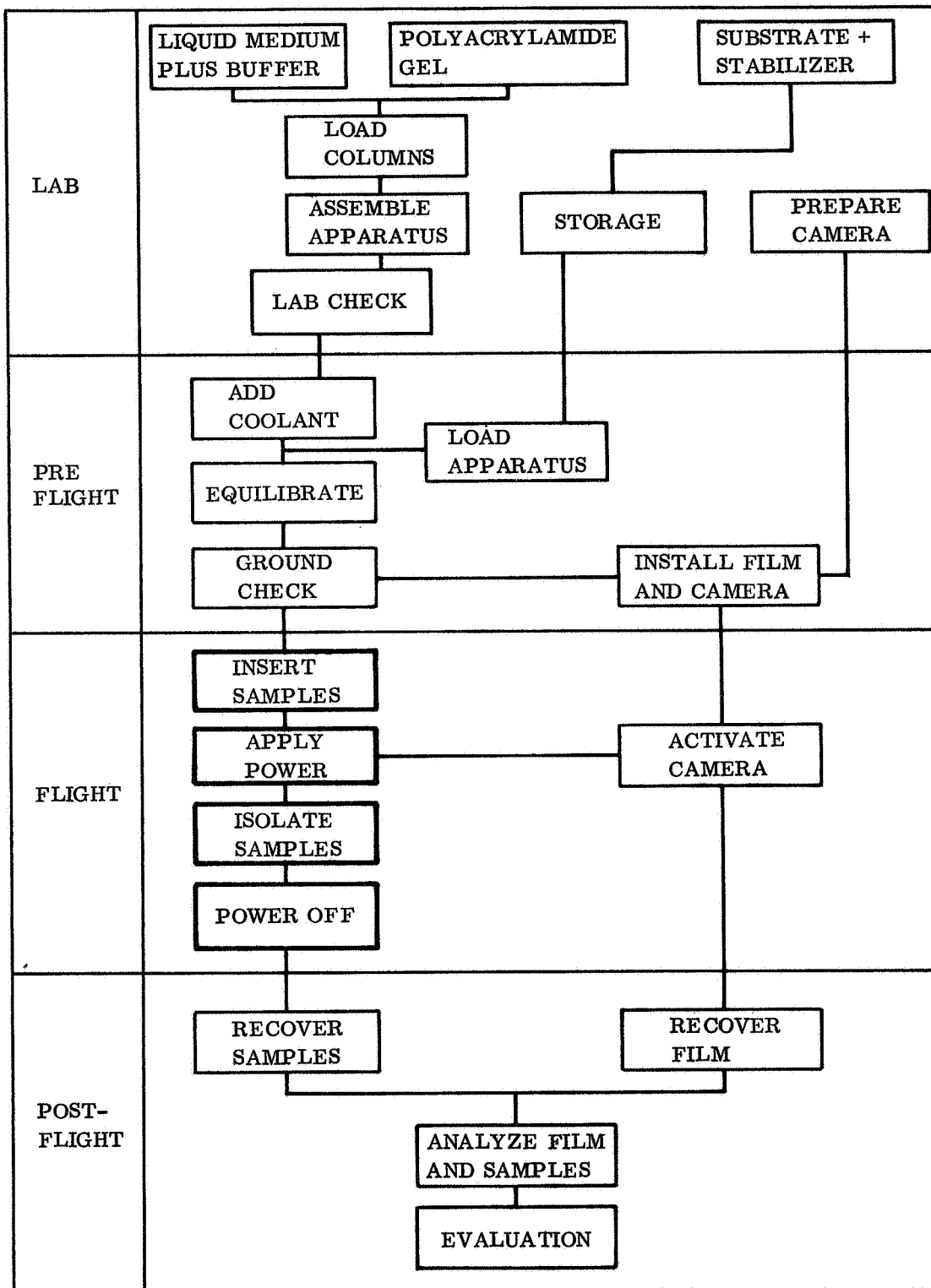


Figure 7.1-2. Process Phase Flow Diagram

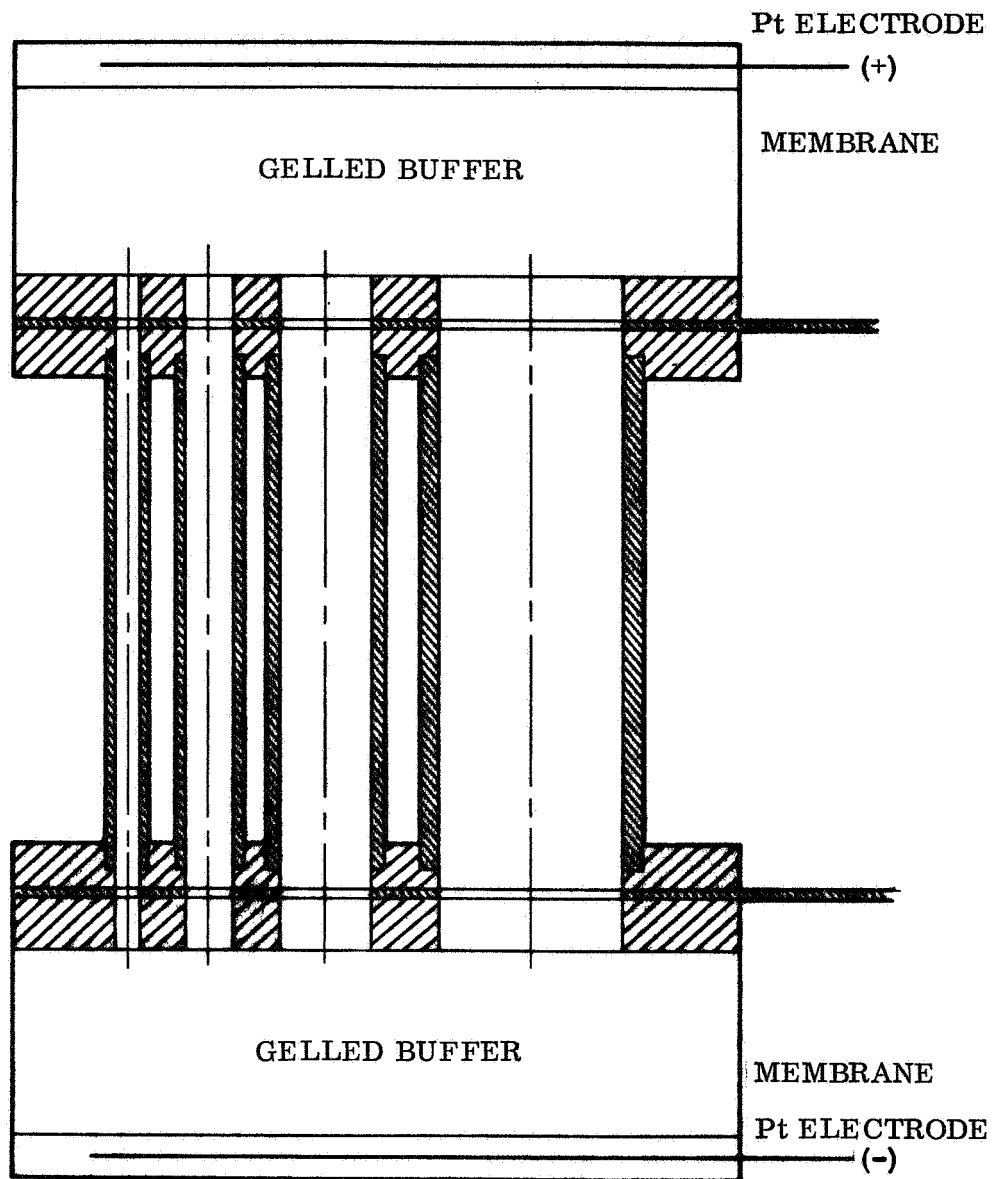


Figure 7.1-3. Electrophoresis Processing Module

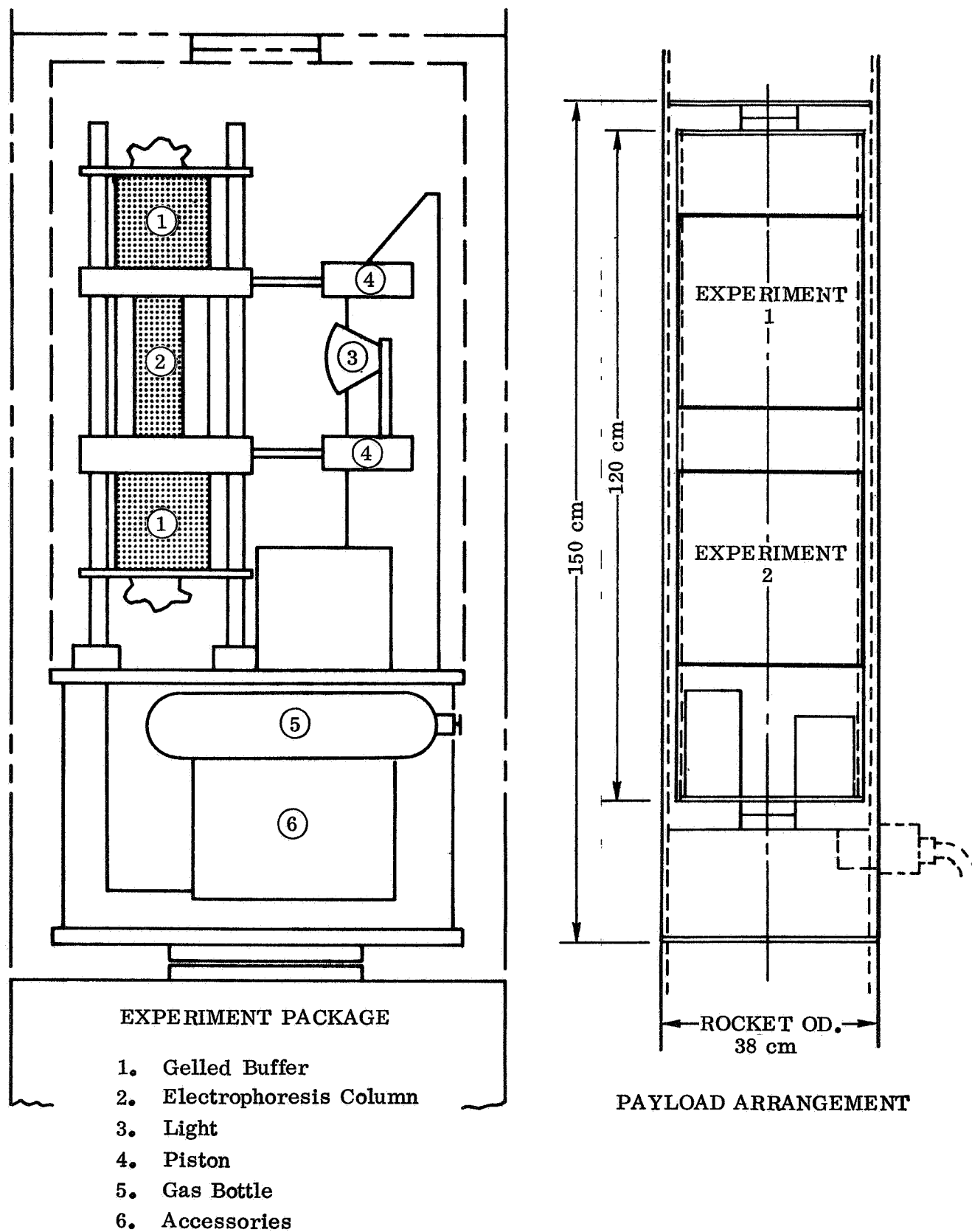


Figure 7.1-4. Electrophoresis Payload

# EXPERIMENT PROGRAM – TRAJECTORY B

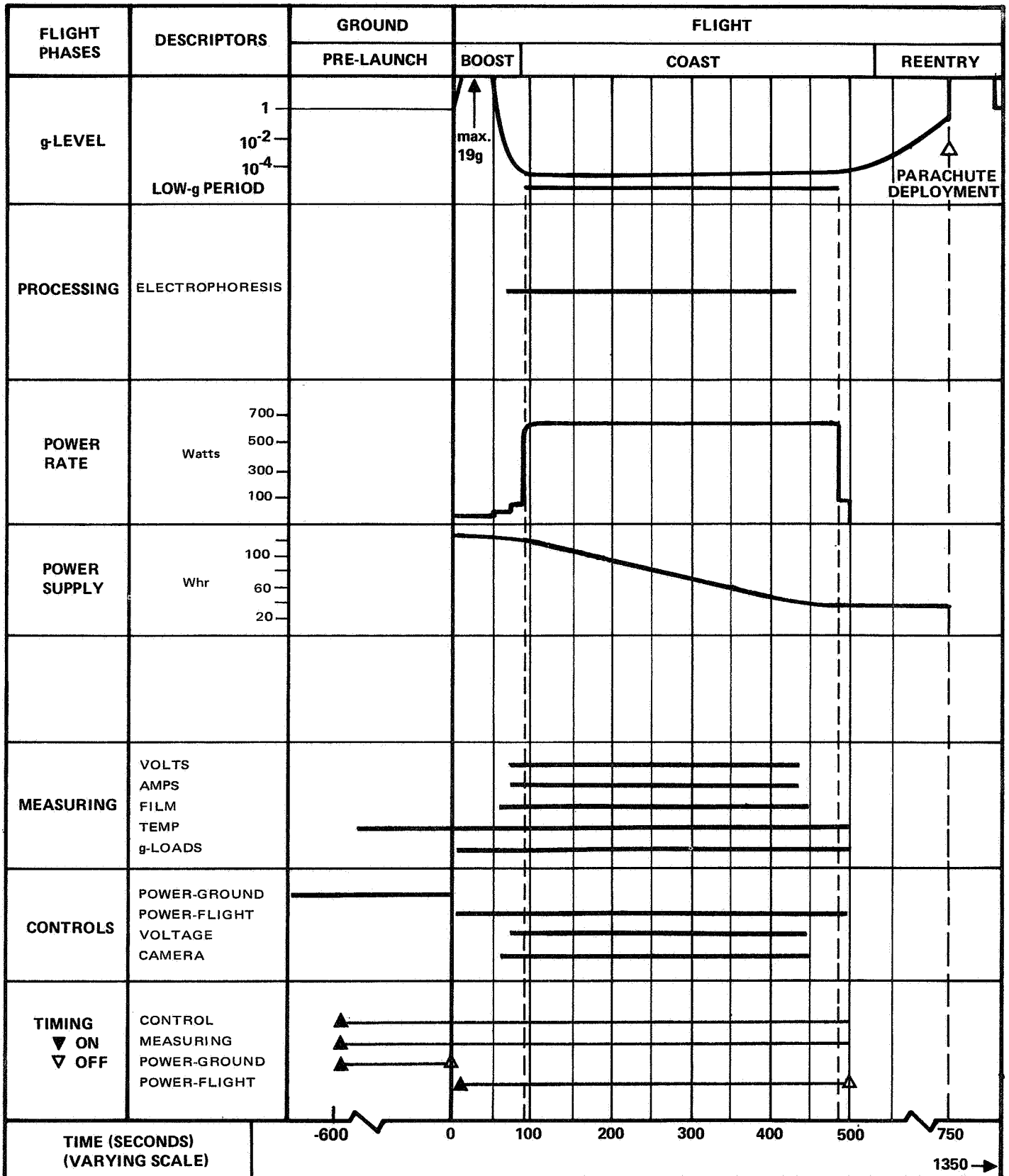


Figure 7.1-5. Time Diagram - Electrophoresis Level I  
Experiment (Aerobee 170 - Trajectory 1)

## 7.2 ELECTROPHORETIC SEPARATION OF BIOCHEMICALS - CONTINUOUS

### 7.2.1 Process Definition and Objectives

Among the important rapidly developing technological advances have been the recent improvements and expansion in electrophoresis and electrophoretic techniques. These have made possible a substantial increase in our understanding of the basis for cell life and many molecular-scale biological phenomena. Diagnostic techniques have been speeded up and many abnormalities and pathological conditions previously difficult to determine are now being readily identified. Electrophoretic techniques have made an especially important contribution in applications to analyses in clinical chemistry.

Although rapid diagnostic tests are now possible, the further extension of the techniques is blocked by some very serious limitations. Among these are:

- (1) Thermal gradients and density gradients are created which tend to generate fluid motions which oppose the separation and isolation motions caused by the electric current.
- (2) The method is not easily adaptable to scaling-up to enable the actual attainment of quantities of material. Relatively large amounts are needed for study and research, for submission to chemical manipulations or for applications in physiological uses.

Electrophoretic procedures have been used in a growing list of laboratory tests and general research problems concerning enzyme and protein abnormalities, serum chemistry, virus and vaccine preparations and isotope separations. A practical and effective method for obtaining these materials in the pure state in quantity would be a large step forward. Electromagnetophoresis has this capability.

Electrophoresis is a process in which (a) a charged particle, moves in (b) a conducting medium, under the influence of (c) an electrical field. Cells and fluids from all living materials are composed of constituents containing acidic ( $\text{-COOH}$ ) and basic ( $\text{-NH}_2$ ) groups which may ionize and thus attract or repel charged particles in the medium. Most material media are conducting or can be made to conduct by the appropriate



addition of salts which dissociate into charged particles when in solution. When electrodes are immersed in the system, the oppositely charged particles move in the appropriate direction toward the electrodes. Particles of colloidal size have an extensive surface. Because of the resultant surface energy these particles tend to absorb ions, especially  $H^+$  or  $OH^-$  from solution. Consequently they are also mobile carriers of charge and move under an applied potential.

Depending on the existing state of a system each component will possess a characteristic equilibrium charge and will move at its own prescribed rate in an imposed electrical field. The mobility depends on the size, shape, net charge, degree of ionization, nature of the conducting medium and the strength of the field. Advantage can then be taken of the different speeds of travel to separate a mixture into its constituent particles and/or ions. Although it is difficult to predict the results under a given set of conditions, empirical determinations may be made to give the most accurate and reproducible results.

The technique has problems and disadvantages. These include stabilization of the boundaries of the migrating species, boundary anomalies and incomplete separation of the charged particles as well as rather extended time requirements. The main experimental difficulty which must be overcome however is control of diffusion and convection currents.

The passage of electricity through the medium produces heat. The heat causes local density gradients which results in motion. This in turn remixes the separated material and reduces the sharpness of the zonal boundaries separating the constituents.

The purpose of the experiment in continuous electrophoresis (electromagnetophoresis) is to use the absence of gravity to avoid the possibility of thermal gradients producing gravity gradients which cause churning and circulatory motions. Some initial successes in this direction were achieved by using a horizontal revolving tube with methylcellulose packing to prevent disturbances. Simpler methods have consisted in incorporation of the solution containing the buffer into some stabilizing medium such as paper, agar, cellulose acetate, acrylamide and similar supports. Although these devices are simple, inexpensive, fast and small in size, they do not lend themselves to preparative methods.

Interaction of the substrate with the support is a decidedly disadvantageous feature. Most stabilizing media have a tendency to absorb proteins and sometimes insoluble complexes are formed. Evaporation of the buffer and inhomogeneities of the support also cause irregular separations.

Electromagnetophoresis is an electrophoretic technique which employs a unique design of a curved apparatus to overcome the problems which arise from the use of support media. The conducting medium is the buffer solution itself. It is trapped in the annulus between two concentric tubes. Charged particles entering the annulus are caused to traverse spiral paths to the electrodes while under the influence of an electrical field, just as the armature of a motor. If all variables of the system are held constant, each particular constituent of a mixture injected into the annulus will reach the same point when it exits the annulus, so that injection, flow and collection can be continuous. The spiral path traversed by the charged particles can be controlled to consist of one turn or more than one turn. The time to make a turn is of the order of one minute. The electromagnetophoresis in low-g is therefore ideal to (a) overcome gravity gradient mixing, (2) allow component collection, (3) allow preparation scale separations and (4) permit separations impossible in one-g.

#### 7.2.2 Verification Requirements

The advantage of zero-g or low-g processing will be determined by the achievement of continuous separation of species in solution through the collection of different species at separate and distinct locations at the discharge zone of the apparatus. The preliminary verification must be that separation occurs and that it is different from the same separation performed in one-g.

To attain the separation and the collection of particles of different species several verification requirements must be met as follows:

1. Processing requirements to avoid establishing environmental conditions which are conducive to remixing currents:
  - 1a constant buffer flow rate
  - 1b constant temperature

- 1c constant current
- 1d constant voltage
- 1e steady zero, or low-g forces.

Also to be verified are:

- 1f separation of distinctive species within a few helical rotations in the apparatus
- 1g known magnetic field strength
- 1h speed of separation.

## 2. Verification of material properties

- 2a purity of species
- 2b resolution
- 2c concentration of substrates (enzyme, etc.)
- 2d pH level of buffer
- 2e viability of cells

The degree of success of the experiment does not depend on in-flight measurements. Property measurements on the recovered material may be made at the termination of the experiment.

Generally, recovery of living cells should not be delayed. The cells will be separated and collected in individual receivers in fractions which differ in mobility. Laboratory analyses on the cells will then be made to verify the properties 1a through 2e which determine the effectiveness of the method in low-g.

Two degrees of test complexity lend themselves to verification at two levels.

### Verification level I:

This experiment will be a "one shot" injection of known, premixed material which will undergo electrophoresis and be separated into constituents which will be caught in individual containers aligned in series. Each container can then be sampled and analyzed by number to determine the properties of the product, and an evaluation made of the separation as compared with the same experiment performed in one-g.

### Verification level II:

A mixture of material from a reservoir will be continuously injected onto the electrophoresis column and separation effected. Measurements can then be made as before

plus quantitation of additional properties for evaluating the effectiveness in collecting the particles of different species on a continuous basis.

### 7.2.3 Experiment Materials

The following criteria are used for the selection of experimental materials:

1. Consist of known practical biologicals.
2. Composed of fractions difficult to process in one-g.
3. Consist of materials which are useful for a preparative-scale and compatible with the objective of large scale production.

The material used for verification level I which will be used to show the different effects produced by one-g and low-g processing. Another set of materials will be used for verification level II experiments. The level I material will be the same as or similar to the substrates used for stationary electrophoresis. These consist of readily separated well characterized dye materials with distinctive color for easy measurement by photographic or other means. The important tests at level I obviously are primarily process verifications, not material verifications. The substrates meeting the criteria are as follows:

<u>Material</u>	<u>Color</u>	<u>Mobility</u>
a. Evans Blue	Blue	Fast
b. Bromphenol Blue	Blue	Fast
c. India Ink	Black	Medium
d. Albumin	(UV)	Medium
e. Rose Bengal	Red	Slow
f. Hemoglobin	Red	Slow

These mobilities are not absolute. They will be used in either of the combinations (a, c, e) or (b, d, f) in which case the relative mobilities will be as indicated.

Much of the work in electrophoresis is concerned with clinical applications in which qualitative methods are used to show the composition of a sample and to identify the components as in level I tests. Less frequently, the components must be separated

and collected, and the collections accumulated for use. This is often difficult because of the convection and sedimentation problems. Human kidney cells from 28-32 week fetuses or from children less than one year old have been shown to be active in producing cells which provide urokinase, a blood-clot dissolving enzyme. This is produced by only 5% of the cells however. These cells cannot be isolated by ordinary electrophoresis because sedimentation occurs. Although the active cells cannot be separated from the bulk cells by ordinary methods, separations have been shown to be possible when convection and sedimentation is avoided. The active material is fairly well known and is extremely practical in circulatory and heart problems. It has excellent probability of success in low-g; and it is required in large quantities.

Although many materials suggest themselves as substrates, the criteria are best met by the following substrate for which demand greatly exceeds supply and is very probably amenable to separation and collection.

<u>Material</u>	<u>Source</u>	<u>End Product</u>
Kidney Cells	Human Fetus	Active Cells

The active cells are afterward subcultured and used to produce urokinase.

#### 7.2.4 Material Quantities, Sample Size

The material quantities must be chosen to accomplish the prescribed purpose. There are two objectives or levels of operation as described in 7.2.2. Verification Requirements: Level I and Level II

7.2.4.1 Level I Quantity. The amount of material required for the Level I experiment is very small. The material is dispersed into a volume of about one ml and each ml contains enough material to provide a vivid color when separated. To accomplish the objective it need only be shown that the particle population is heterogenous, and that separation is attained, by collecting the separated species in different receivers. The amount of suspended particles injected onto the column can be contained in about 0.05 ml and be injected at the rate of approximately 0.5 ml/hr.

7.2.4.2 Level II Quantity. The material quantity required for verification Level II is the amount which can flow for 300 seconds at the rate of 0.5 ml per hour and contains approximately  $4 \times 10^7$  cells per ml.

Sufficient buffer must be used to fill the buffer compartments and the buffer reservoir with enough liquid to last the course of the experiment. For flight hardware, this may be accomplished with about 10 liters of solution. The buffer agent is chosen with regard to the experiment substrate. "Hydrion" buffer tablets may be used to attain a pH of about 7.0 for Level I experiments. A more sophisticated buffer with closer pH control is needed for Level II experiments. These require a "Tris" buffer of the following composition for each liter of solution:

10g Tris-(hydroxymethyl) aminomethane

1.4g Ethylenediaminetetraacetic acid-disodium salt

0.8g boric acid

This buffer has a pH of 7.4 and molality of 0.012.

Ordinary ambient water may be used for cooling but considerable improvement in the sharpness of streaks is secured by filling the tank containing the coolant with ice water. This requires a total of 6.0 liters of water-ice mixture to cool the prechilled electrode compartments, buffer compartments, cell compartment and to allow some "hold" time.

#### 7.2.5 Experiment Process Phases

The experimental process phases for continuous electrophoresis are similar to the procedures for stationary electrophoresis. There are more manipulative procedures however because of the greater complexity of the apparatus. For this reason, a liberal time for the pre-test phase is necessary to assure that flow rates are precisely adjusted. The adjustments may be made on the ground after which they remain fixed for all runs. The processing consists of the following steps:

(1) Prepare substrates

(2) Prepare buffer [pH(Tris), ionic strength ( $B_2O_3$ ), density (sucrose)]

- (3) Prepare coolant (ice slush)
- (4) Adjust inflow and outflow rates
- (5) Apply power; circulate coolant
- (6) Inject samples
- (7) Collect samples
- (8) Remove product
- (9) Evaluate results

The sequence of operations divides itself into four separate steps as indicated below.

7.2.5.1 Pre-test laboratory operations consist of sample preparation and setting flow conditions in the following steps:

- (1) Mix buffer
- (2) Mix dyes and buffer (or kidney cells and buffer)
- (3) Fill cell compartment, buffer compartment and electrode compartment with buffer
- (4) Fill buffer supply reservoirs with buffer
- (5) Expel all gases in liquid compartments and prepare for sample injection
- (6) Adjust inlet and outlet flow conditions in all compartments
- (7) Apply current and check sample (dye) flow and sample receiver operation with coolant circulating in system
- (8) Set all adjustments for standby operation

Because flow control and steady-state conditions of current or voltage are critical to the success of the experiment, these variables must be positively and reproducibly preset in the laboratory. Temperature control is also important because of its effect on conductivity. Therefore the coolant flow must also be adequate to maintain a consistent temperature, preferably 0° C. Not only is this particular low temperature convenient to maintain but the low temperature is better because it improves the sharpness of the streaks of product fractions.

7.2.5.2 Pre-test (ground) operations consist of the following steps

- (1) Install apparatus, camera, UV
- (2) Load buffer and coolant solutions
- (3) Check and confirm flow conditions
- (4) Thaw and mix sample material with buffer solution. Equilibrate temperature.
- (5) Start coolant flow
- (6) Check energy flow and adjust if required

It is essential that the flight begins with a chilled and stable system with the numerous inlet and discharge flows balanced and steady.

7.2.5.3 Test (low-g) operations consist of the following events:

- (1) Begin coolant flow (if standby cooling has been interrupted)
- (2) Apply constant electrophoretic power
- (3) Inject sample onto column
- (4) Collect fractions

It is important for this phase that the operating conditions be positively and accurately stabilized in the pre-test phase. Otherwise the product may not be collected at the desired discharge tube. It is also important that the power supply consist of a constant current or constant voltage source so that the rotational speed of the charged particles does not vary.

7.2.5.4 Post-test operations consist of recovery of samples and evaluation of the product in each of the receiver tubes. Comparison is made with one-g resolution of dyes and the amount and fraction for which fibrinolytic activity is present in kidney cells. Alternatively or simultaneously, color film analysis of the dye stream and UV absorption of colorless materials are evaluated.



7.2.5.5 The processing sequence is identified in the flow diagram Fig. 7.2-1 and the preliminary time diagram Fig. 7.2-2.

#### 7.2.6 Low-g Test Requirements

7.2.6.1 Low-g Time Requirements . It may be shown that if no forcing conditions are used the time required for an ion to make one revolution in the endless belt apparatus is about 10 seconds as follows:

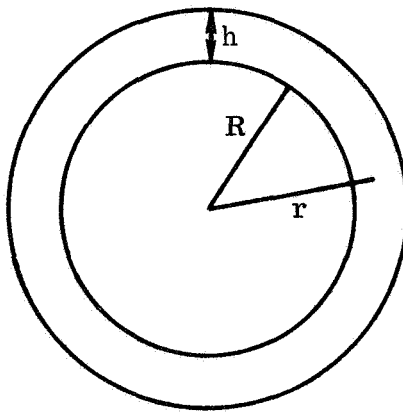


Fig. 7.2-3. Transverse Section of an Annular Tube

The figure shows a transverse section through an annular tube. If the width of the annulus is small compared to the mean radius of curvature, Lamb showed that the rotary flow of the fluid can be treated as a viscous flow between straight parallel plates.\*

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\*H. Lamb, Hydrodynamics, Dover Publication, N.Y., N.Y., 1945, p. 582.

The inner radius is  $R$ , and the outer one is  $R + h$ . The distance of an arbitrary point from the cylinder axis is  $r$ . The local tangential velocity  $u$  at radial distance  $r$  due to rotary motion of the fluid in the annulus is given by

$$u = \left(\frac{1}{2} \eta\right) z (z - h) \, dp/dx \quad (1)$$

where

$$z = r - R$$

$$\eta = \text{viscosity}$$

$$dp/dx = \text{tangential pressure gradient}$$

The maximum velocity at the center of the annulus is obtained by setting  $z = h/2$ :

$$u_o = -h^2/8\eta \, (dp/dx) \quad (2)$$

If a magnet of flux density  $B$  is in the center of the tube and the current density from end to end in the annulus is  $J$  then the value of  $dp/dx$  is given by:

$$-dp/dx = f = \left(\frac{1}{10}\right) [J \times B] \text{ dynes/cm} \quad (3)$$

if  $J$  is in amps/cm<sup>2</sup> and  $B$  is in gauss.

Combining (2) and (3) we obtain:

$$u_o = (h^2/80\eta) [J \times B] \quad (4)$$

If we confine our attention to a thin streak of charged particles injected at the center of the annulus, we can limit ourselves to the spiral path described by an ion midway between the walls. The period of revolution,  $\tau_o$  for this central fluid layer is calculated from (2) as:

$$\begin{aligned} \tau_o &= 2\pi/w_o = (1/u_o) 2\pi(R + h/2) \\ &\cong 2\pi R/u_o \end{aligned} \quad (5)$$

From (4) and (5) it follows that

$$\tau_o = \frac{160\pi R \eta}{h^2 [J \times B]}$$

If we assume  $R = 1.5$  cm;  $\eta = 10^{-2}$  poise;  $J = 10^{-2}$  amps/cm<sup>2</sup>,  $h = 0.15$  cm;  $B = 600$  gauss, it follows that

$$\tau_o \cong \frac{160\pi 1.5(0.01)}{.02(0.01)(600)} \cong \frac{7.20}{.12} \cong 60 \text{ sec}$$

This value may be readily increased or decreased by changing the central permanent magnet or by varying the current density by means of voltage changes. It is evident therefore that the experiment lends itself well to the zero-g capability of rocket flights but is severely limited if the tests were to be made in a drop tower. Drop tests would not be out of the question but would require some stringent operating requirements to permit separations to be made within 4 seconds of low-g time.

To obtain a single revolution in a maximum of 4 seconds would require a very small  $R$  and  $\eta$  and a very large magnetic flux and electrical field. It can be shown that resolution is seriously degraded if  $h$  is made large.  $R$  might be reduced by half but at the expense of annular thickness (because of wall friction).  $\eta$  cannot be varied much as most electrolytes have a viscosity of  $1 \times 10^{-2}$  poise, or more.

A combination of strong magnets and high current density can be used for short time periods as energy sources in an endless belt experiment. However, the apparatus is not amenable to the required miniaturization. Electrolytic gassing from the high current density might also be a problem. It is therefore considered that a rocket provides the low-g time required to perform the electromagnetophoretic separation, but not a drop tower.

The time required to resolve two streams of different mobility may also be calculated. For example, Kolin and Luner<sup>\*</sup> have run hemoglobin-albumin mixtures on the endless belt machine. They found current to affect the apparatus temperature as follows:

<u>I, in A</u>	<u>E, V/cm</u>	<u>T<sub>meas</sub>, °C</u>	<u>T<sub>max</sub>, °C</u>
150	68	16.5°	24°
200	73	23.6°	35°
250	85	31°	43°
300	90	39°	54°

\*A. Kolin and S. J. Luner, "Continuous Electrophoresis in Fluid Endless Belts," Anal. Biochem 30, 111 (1969)

The electric field increases as the current and temperature rise. The maximum temperature halfway between the fluid core and the mouth was calculated on the basis of a parabolic distribution of temperature. From these results the maximum current for biological work was limited to 200 mA. Other parameters were as follows:

Buffer flow rate	2.5-4 ml/sec
DC potential	1 KV, constant
Period of revolution	35-40 sec
Collector feed rate	0.6 ml/min
Injection feed rate	0.002-0.025 ml/min

Figure 7.2-4 shows the separation between streaks of albumin and hemoglobin when electrophoresed under the above conditions. If injection occurs at one side of the belt and product is collected 180° later at a path length of about 10 cm the figure shows that a separation of about 2.6 mm in the streaks of the products has been effected. This requires 15-20 seconds of travel under steady conditions.

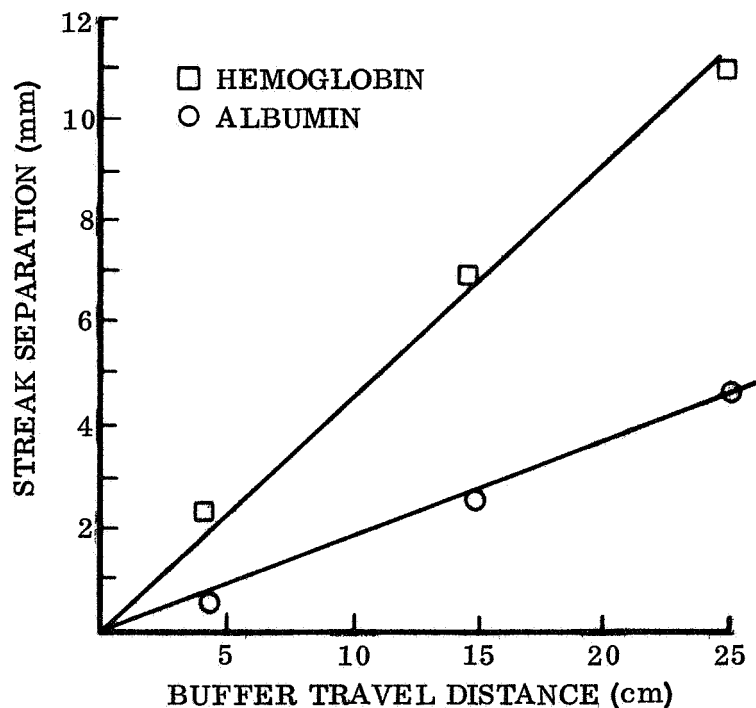


Figure 7.2-4. Separation of Particle Streaks vs Distance of Travel

Good separation with less possibility of mixing the fractions in the collection tubes would be attained in another revolution of  $360^{\circ}$  to give a distance between streaks of about 7.8 mm. These streaks are easier to collect without mixing and require 100-120 seconds to make the trip. The mobility difference of the particles under the conditions of the experiment was  $3.8 \mu/\text{sec}$  per volt/cm field strength.

For verification level I for which correlations with one-g experiments are made, a single revolution for stabilization plus photo measurements of the separation distance of bromphenol blue and rose Bengal would suffice for an evaluation. The low-g time required would be a minimum of about 4 seconds and an average of 35-40 seconds. To this would be added time for stabilization of one revolution or 30 seconds. Therefore a minimum low-g time for a qualitative evaluation of the continuous electrophoresis experiment is about 35 seconds. Quantitative measurements could be made for low-g periods extended beyond this period by collecting and measuring the amount of the individual fractions as well as determining the paths of the respective particles.

The power requirements may be readily calculated also. Constant current or constant voltage power is required. Both will not remain constant if the temperature in the channel rises as current passes. This changes the conductivity. Temperature control is therefore very important to minimize the temperature differences in the narrow annulus and to reduce to temperature level so as to sharpen the separation streaks. Normally, operating conditions for verification level I are 1-1.2 KV at 250 mA or approximately 300 watts. If 200 seconds of low-g time is available, all of it will be used. The power required is therefore 60,000 joules or about 18 watt-hour. A cooling pump will require an additional 2 watt hours.

The power required for verification level II is the same as for level I except that the necessary operating parameters will have been determined from level I experiments. The time for performance can be extended without limit on a continuous basis. The power per hour will be  $1.2 \text{ KV} \times 250 \text{ mA} \times 1 \text{ hr} = 300 \text{ watt hr}$ . If all the energy is converted to heat, this represents  $300 \times 8.6 \times 10^2$  or  $2.6 \times 10^5$  cal. This requires 3,300 gms of ice for cooling or about 9.0 lbs of ice with allowances. Mechanical refrigeration would be used for continuous processing.

The processing times and power requirements for verification level I and II are summarized below.

<u>Sample</u>	<u>I</u>	<u>II</u>
Low-g time, sec	390	390
Processing power rate, watts	300	300
Circulating pumps, etc., watts	100	100
Total energy Wh	59	59

7.2.6.2 Required g-Level. The g-level which is necessary for the performance of electrophoresis has no strict cut-off level. All material is confined in the apparatus. The evaluation depends on obtaining a substantial enhancement of separation in low-g because of lack of thermal and gravity gradients. On the basis of past experience and expected behavior the following maximum g-levels have been defined:

Verification level I:

A steady g-level of  $1 \times 10^{-4}$  maintained during the course of the experiment. The steady state is as important as the level.

Verification level II: A steady g-level of  $10^{-5}$  is desired but an unvarying steady state is as important as the level.

7.2.7 Low-g Test Facilities and Experiments

7.2.7.1 Correlation of Experiment Requirements and Facility Capabilities. A comparison of the time required with the low-g test facilities available show that level I experiments can be carried out in a research rocket class 4 and the maximum standard WSMR trajectory B. (Weight and Dimensional Data from Section 7.2.8):

	<u>Low-g Time</u>	<u>Weight</u>	<u>Height</u>
	(sec)	(kg)	(cm)
RR-4 Limitations	390	125	150
Level I Experiments	390	90.5	125
Level II Experiments	390	90.5	125

The low-g time available is compatible with the requirements outlined in Section 7.2.6. Most of the equipment is made of plastic and is very light. The largest amount of weight is attributed to buffer and coolant solution. Power supply and pumps are responsible for the remaining weight which totals well within the rocket capabilities. The apparatus is capable of continuous uninterrupted service. Quantity production on a large scale can be accomplished with the same size and weight apparatus (except for the battery) if arrangements are made to replenish the buffer concentration. Such large scale production requires long zero-g times. A vehicle like Skylab is necessary for such purposes.

#### 7.2.8 Experiment Apparatus and Payload Definition

The basic apparatus is shown schematically in Fig. 7.2-5. It is composed of three fluid systems:

1. Buffer supply system
2. Coolant water system
3. Sample injection and receiving system

The walls of these systems are compartmentalized into three chambers:

1. Electrode chambers (two)
2. Buffer chambers (two)
3. Electrophoresis chamber

An electrical system provides motive power.

7.2.8.1 Apparatus Description. The buffer supply and receiver reservoirs  $R_s$  and  $R_r$ , Fig. 7.5-5(a) are completely filled with buffer and the system is purged gas-free on the ground. Flow control is provided by main valve  $V$  and control valves  $v$ , as well as restricting valves  $vr$ . These valves are adjusted to perform the following functions:

1. Furnish a conductive medium.
2. Purge electrode compartments  $E_1$  and  $E_2$  of evolved gases and oxidation-reduction products.

3. Control the flow of buffer between left and right buffer compartments so as to provide the desired fluid transport between buffer compartments  $B_1$  and  $B_2$ . Total buffer flow is about 2.5 to 4 ml per second.

The sample injection system contains a control valve  $V$ . Sample flows into the electrophoresis chamber at the rate of 0.002 to 0.025 ml per minute. The sample system is charged with buffer prior to the start of the experiment but the fraction collectors  $fc$  are in the unloaded position. As sample is collected, the pistons in the receivers expand to allow room for the product fractions which are diluted by the buffer medium.

The buffer compartments  $B_1$  and  $B_2$  are hydraulically isolated from the electrode compartments  $E_1$  and  $E_2$  containing ring shaped platinum electrodes designated by symbols  $el_1$  and  $el_2$  by semipermeable membranes  $mb$ .

The electrolyte in the electrode chambers is continuously renewed. Not shown is a baffle arrangement which allows the selective outflow of ions produced by electrolysis. A ring of plastic foam,  $a$ , permits electric and hydraulic communication between the fluid belt and the buffer compartments but prevents thermal transport across the boundary.

The fluid belt  $fb$  Fig. 7.2-5(c) is formed by concentric plastic tubes machined to form an annulus of about 1 mm width.

Four magnets, N-S are installed with similar poles oriented head-to-head. When a soft-iron core,  $m$ , is inserted between the poles and within the sample compartment as shown in the figures  $a$ ,  $b$ , and  $c$ , a uniform permanent magnetic field as shown in  $d$  is set-up.

The electrical field is at right angles to the magnetic field when power is applied to the electrodes. Charged particles and ions then describe circular motion within the annulus under the conditions established and are transported to the electrode of opposite charge as well. Since all charged particles and ions possess a mobility unique to themselves, this affords a separation method if collecting reservoirs are placed at the proper location. This is accomplished by the collector, Fig. 7.2-5(a), which contains a series of orifices uniformly spaced. Each orifice contains a tube which leads to the fraction collector,  $fc$ .



7.2.8.2 The Rocket Apparatus Assembly is shown in Fig. 7.2-6 and the payload assembly in Fig. 7.2-7. The data for the major components and the assembly are as follows:

	<u>Weight (kg)</u>
EMP Unit	15
Buffer Supply	3
Cooling System with Coolant	6
Gas Supply System	4
Direct Power Controls	1
Camera with Lights	6
UV Detector/Recorder	1.5
Support Structure	1.5
	<hr/>
Total Apparatus Weight	38 kg
Total Height of Apparatus	55 cm
Power Rate (Apparatus only)	400 watts
Experiment Power Consumption	59 Wh

7.2.8.3 Rocket Support Module. The outfitting and weight of the support module are as follows (numbers in parenthesis identify components specified in Section 5.2.2 and Table 5-1):

Basic Structure (1, 23)	33 kg
1 Battery Pack (4)	3
Power Conditioning (5A, B, C-1, D)	10.5
Programmer, Recorder, Miscellaneous (6, 7, 8)	6
	<hr/>
Total Weight	52.5 kg
Axial Height	55 cm
Power Rate (Instrumentation)	150 W
Power Consumption/Flight	37 Wh

7.2.8.4 Rocket Payload. The complete payload is illustrated in Fig. 7.2-7. Major payload data are:

Payload Weight

Apparatus	38.0 kg
Support Module	<u>52.5 kg</u>
Total Weight	90.5 kg
RR-4 Capacity	125.0 kg
Contingency	34.5 kg

Payload Space (Axial Height)

Apparatus	55 cm
Support Module	<u>70 cm</u>
Total	125 cm
RR-4 Capacity	150 cm
Contingency	25 cm

Payload Power Consumption

Experiment	59 Wh
Support	<u>37 Wh</u>
Total	96 Wh
Contingency	14 Wh

7.2.9 Experiment Performance

7.2.9.1 Pre-test (Ground Operations). The experiment can be prepared as described in Sections 7.2.5.2 and 7.2.6.1. The following operations remain at the launch

- (11) Load samples
- (12) Circulate refrigerant
- (13) Check gas, power, and electrical circuits
- (15) Confirm buffer compartment flow rates
- (16) Confirm absence of gas bubbles
- (17) Load, install and check camera, lights, film and UV detector operation.

7.2.9.2 Test (Flight) Operations. At rocket cut-off, the following operations are programmed:

- (21) Actuate camera
- (22) Actuate buffer flow
- (23) Energize electrophoresis power
- (24) Actuate sample injection
- (25) Actuate UV system
- (26) Stop current and close valves
- (27) Stop camera

7.2.9.3 Post Test (Ground) Operations.

- (31) Remove and store sample fractions
- (32) Recover film
- (33) Drain and renew buffer

7.2.9.4 Time Diagram. The sequence of experiment phases and events is shown in the detailed time diagram, Fig. 7.2-8.

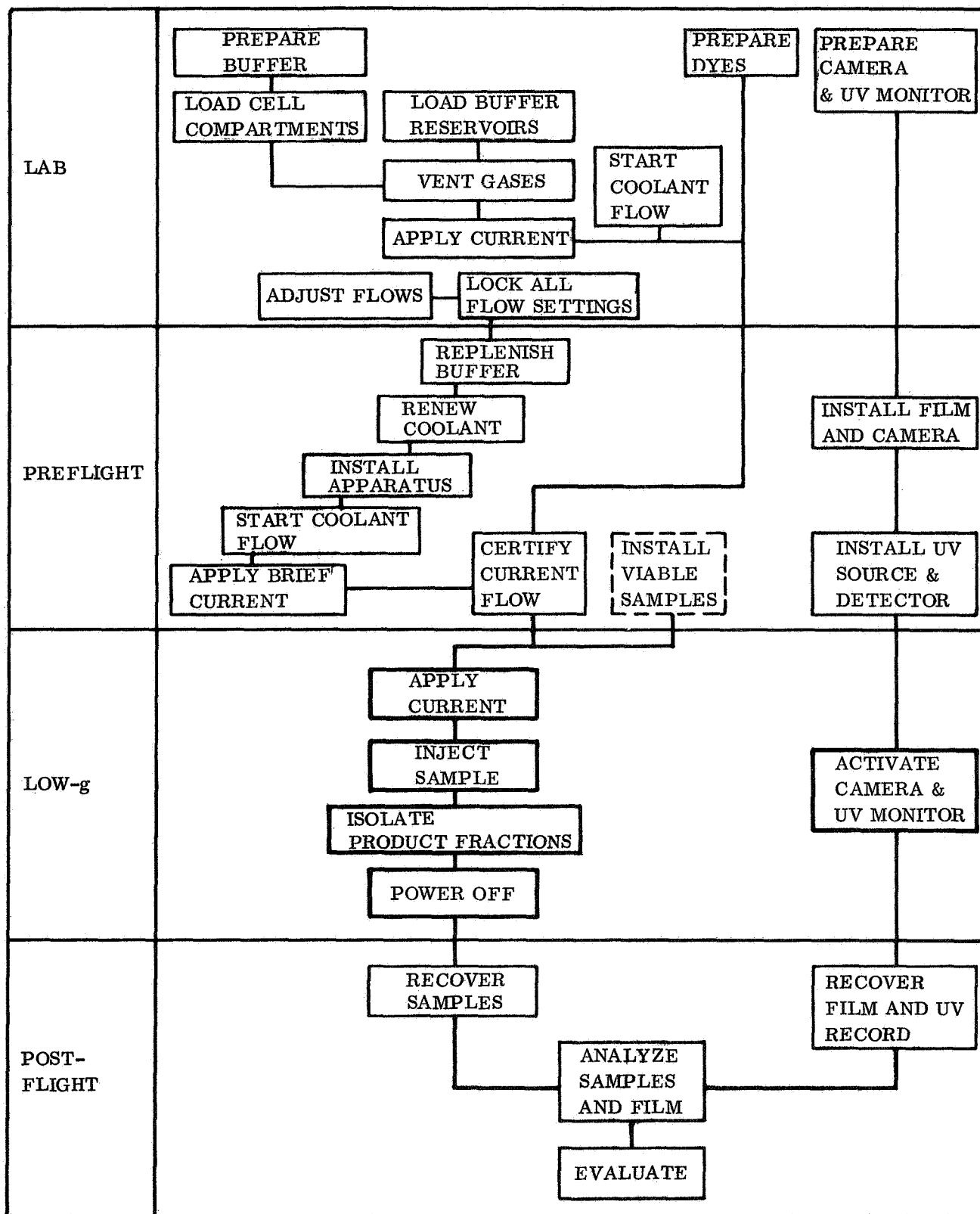


Figure 7.2-1. Flow Diagram for Continuous Electrophoresis











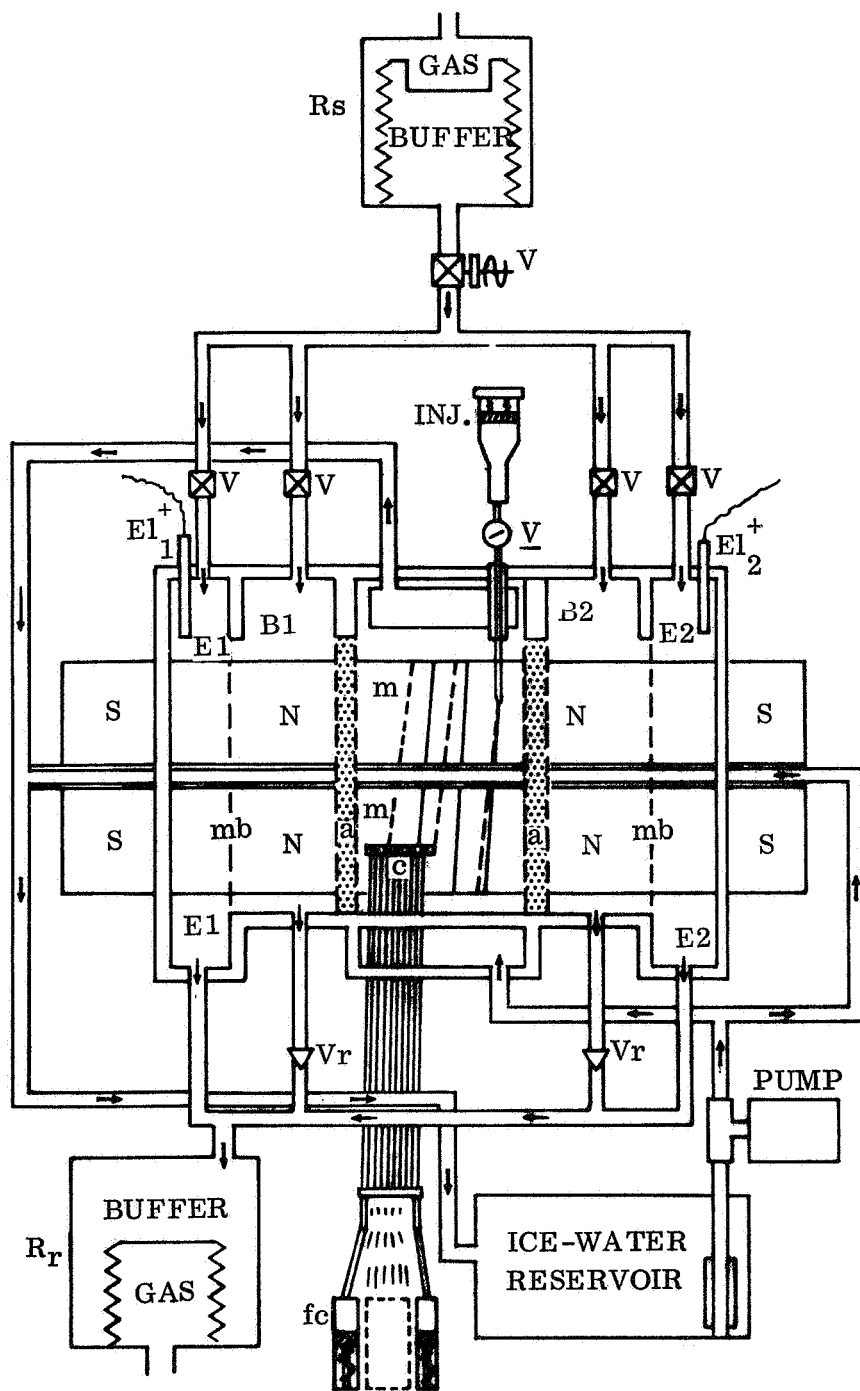
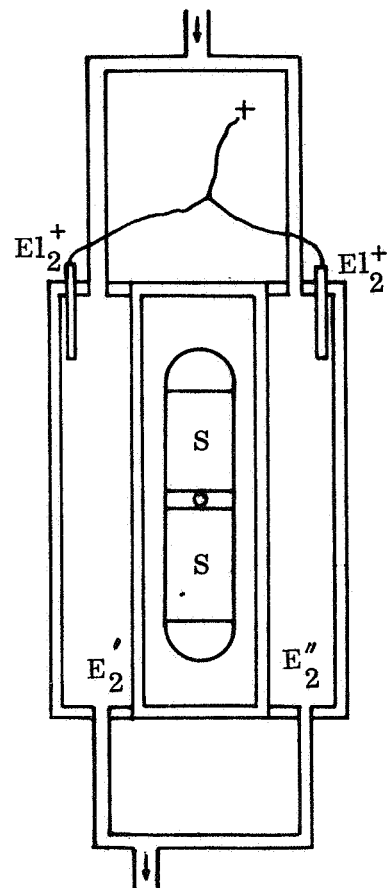
TOTAL FLIGHT TIME	
PRELAUNCH	PREPARE SAMPLES, BUFFER SOLUTION; BEGIN FLOWS, POWER; LOAD EXPERIMENT.
LOW-g TIME	
ENERGIZE POWER	
FLOW COOLANT	
TURN ON CURRENT	
INJECT SAMPLE	
STOP MOTION MOVIE	
UV MONITOR	
ISOLATE PRODUCTS	
CURRENT OFF	
POWER OFF	

Figure 7.2-2. Preliminary Time Diagram



(a) Main Apparatus



(b) ELECTRODE COMPARTMENT

Figure 7.2-5

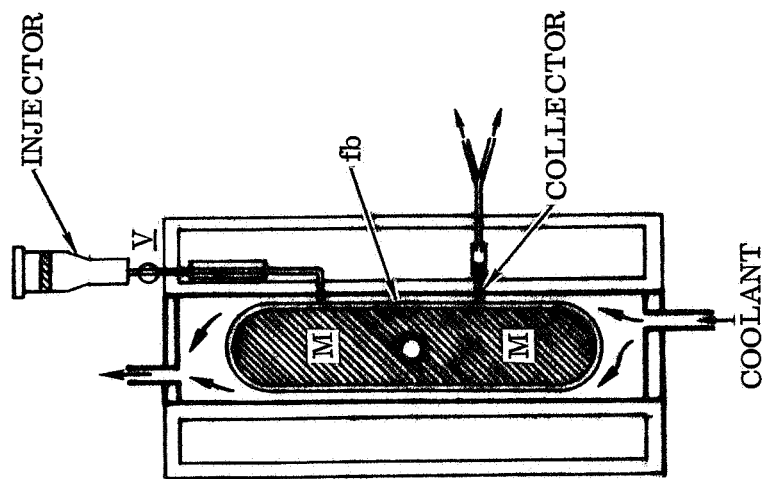
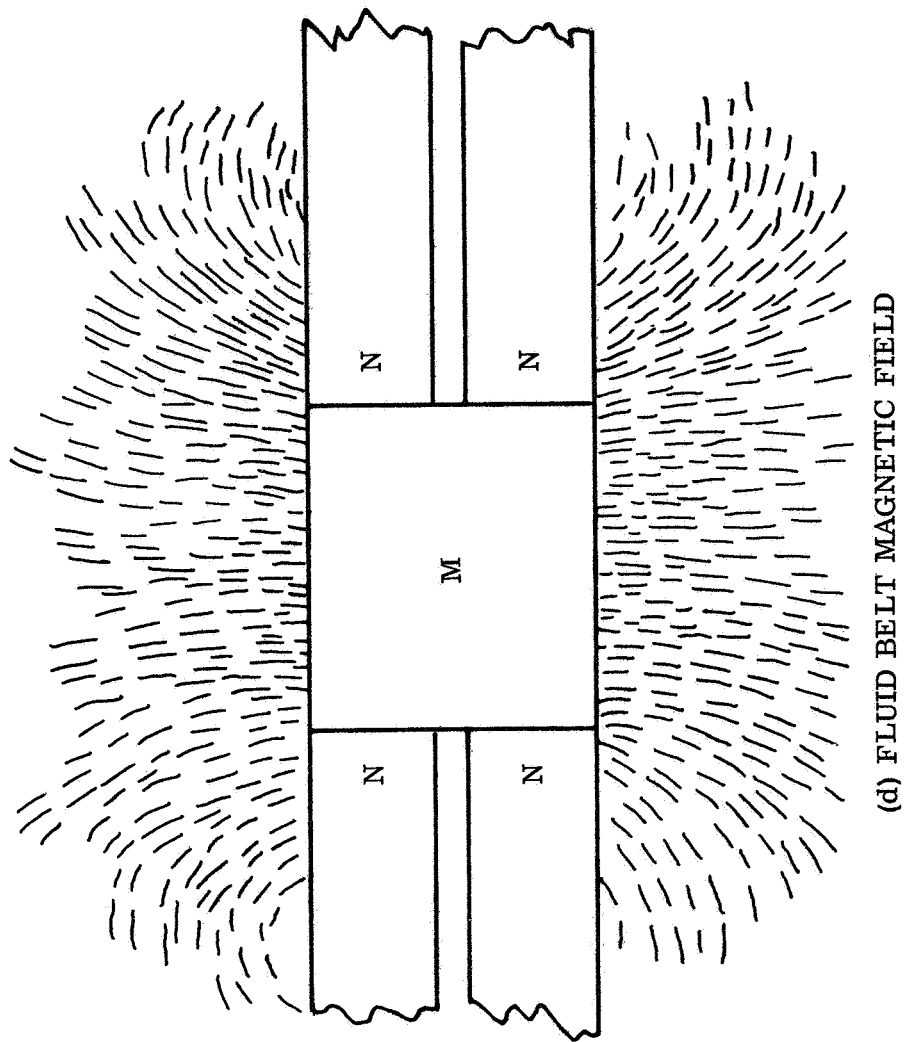


Figure 7.2-5. (Page 2 of 2)

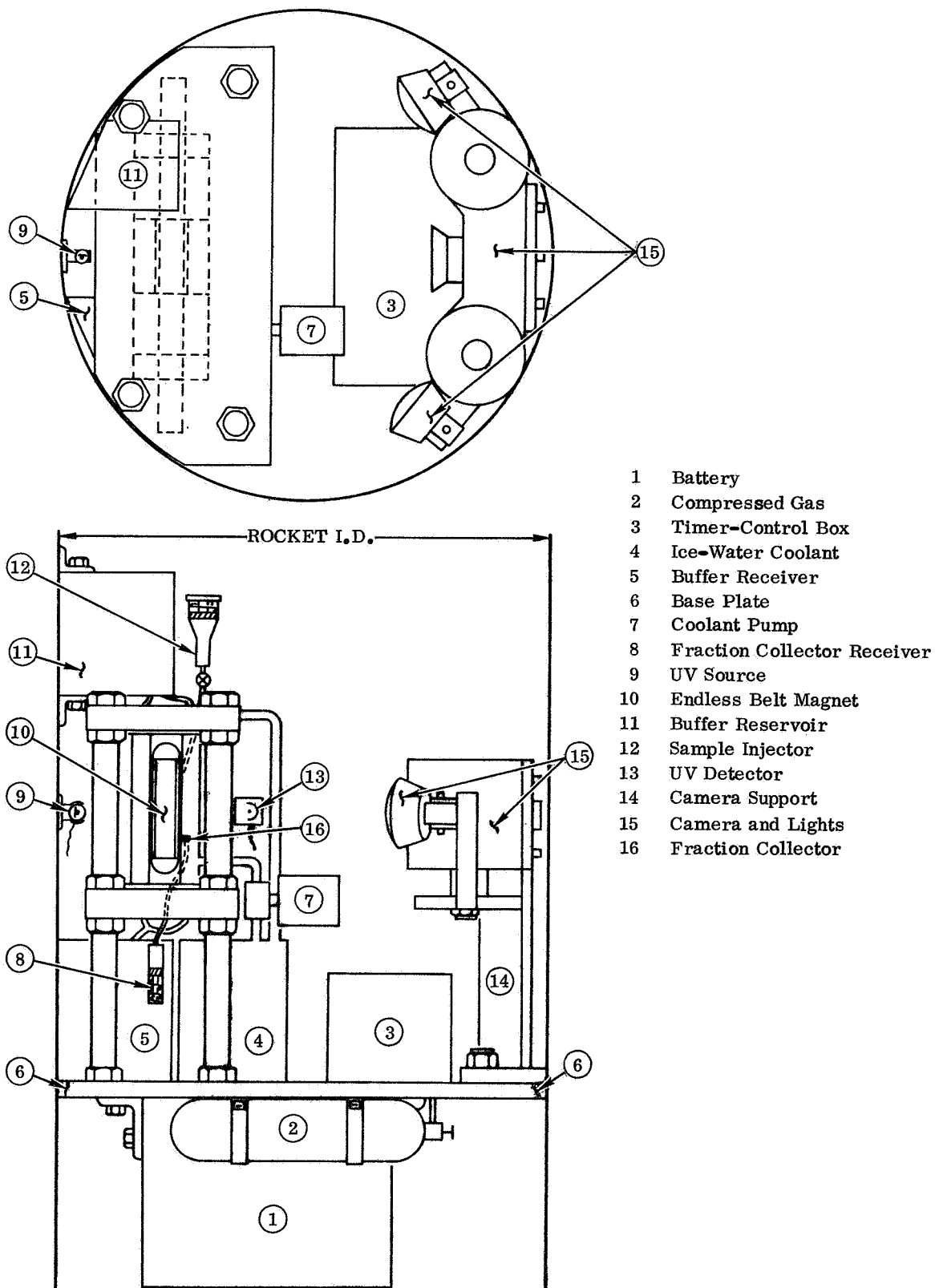
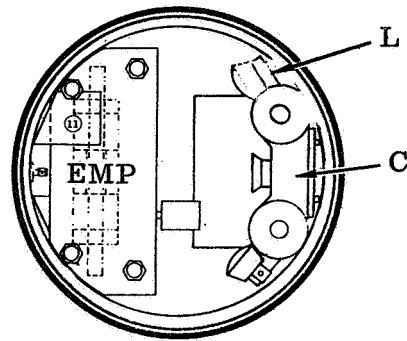
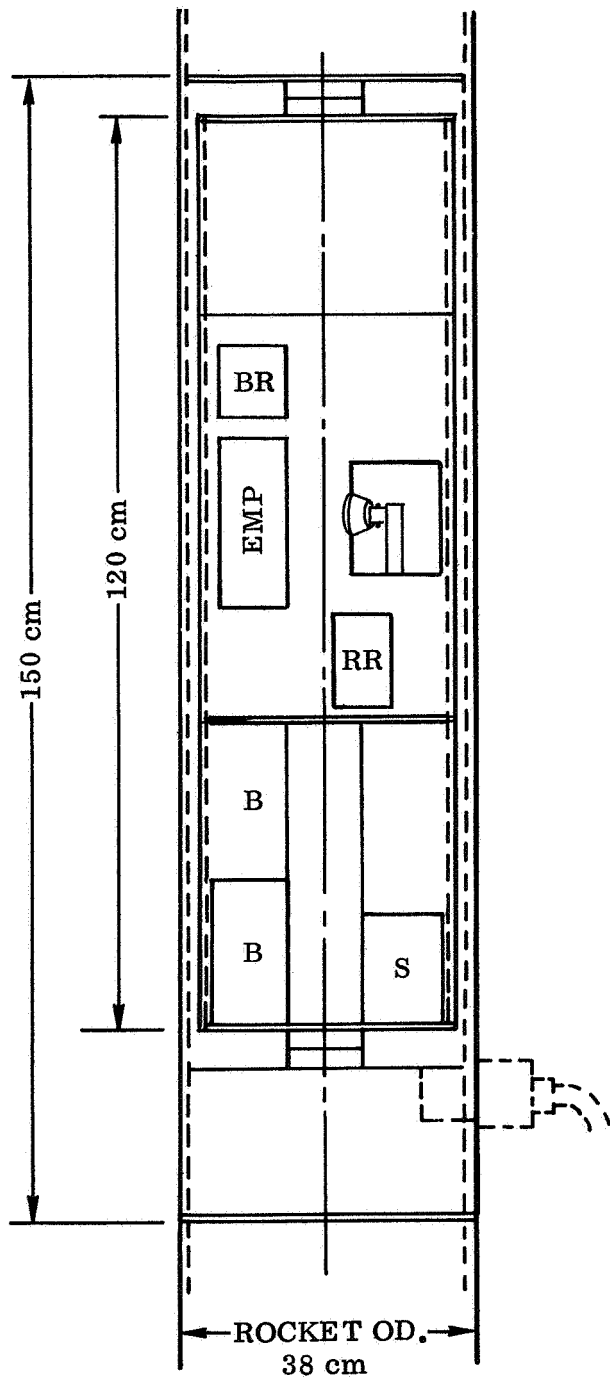


Figure 7.2-6. Assembly, Continuous Electrophoresis (EMP)



# ROCKET PAYLOAD ASSEMBLY



- B - BATTERY
- T - TRANSFORMER
- S - SEQUENCER TIMER
- BR - BUFFER RESERVOIR
- RR - RECEIVER RESERVOIR
- C - CAMERA
- L - LIGHTS
- EMP - APPARATUS MODULE

Figure 7.2-7. Payload Assembly, Continuous Electrophoresis

# EXPERIMENT PROGRAM – TRAJECTORY B

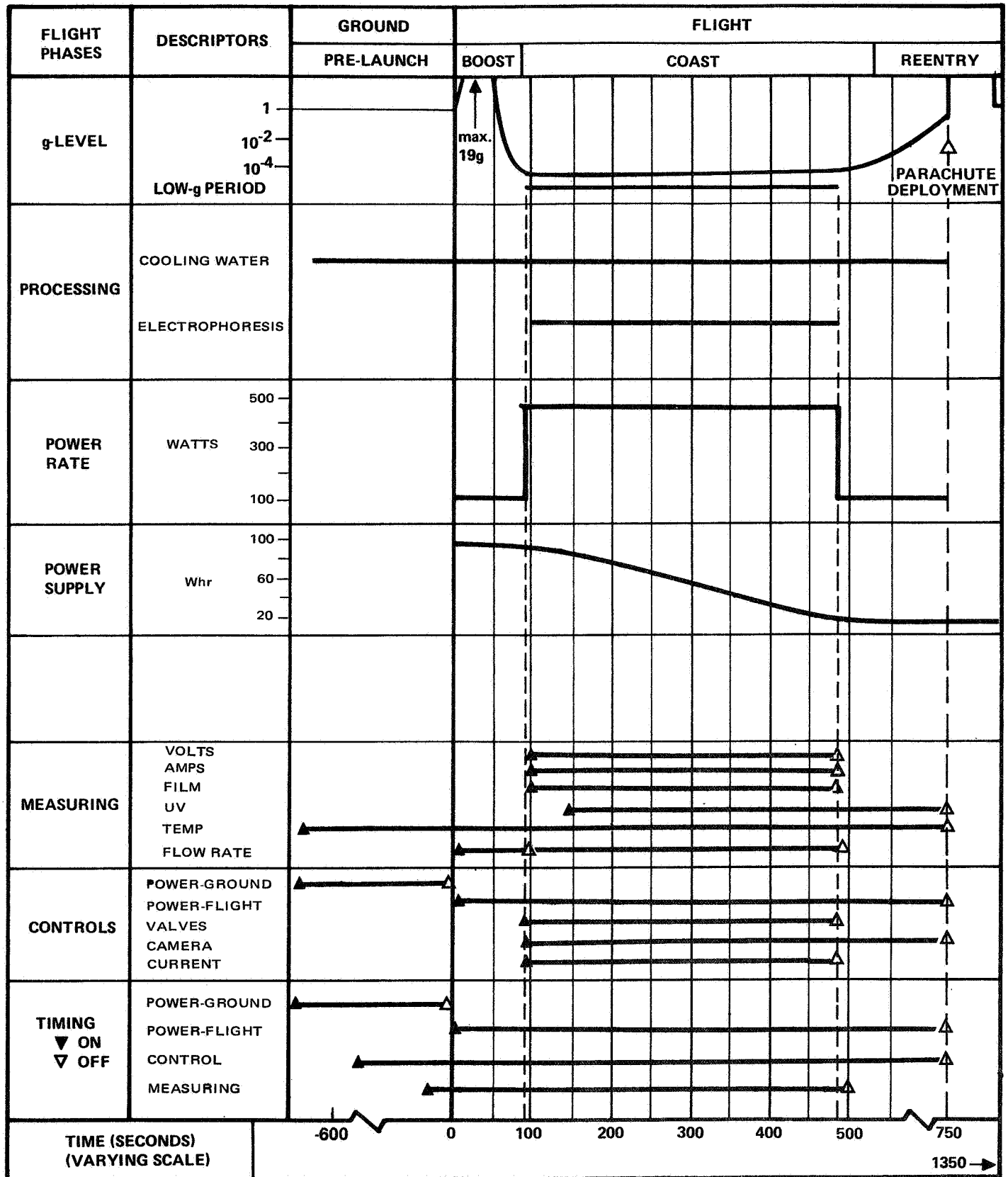


Figure 7.2-8. Time Diagram, Continuous Electrophoresis

### 7.3 FIBER/PARTICLE COMPOSITES/PREDISPERSED

The preparation of random-distributed fiber and particle composites comprises two primary processing phases: (1) the establishment of the reinforcement dispersion and (2) the maintenance of this dispersion in the liquid state and through solidification. Common to both phases is the complex interaction between reinforcements, and between reinforcements and matrix which cannot be determined in the one-g environment. It is particularly complex if the dispersion is produced by liquid-solid mixing (1). This further requires the development of high-temperature mixing techniques and the related equipment. It is, therefore, indicated to reduce the number of variables and problems in initial experiments by the use of a sample in which the reinforcements are pre-dispersed. Experiments with pre-dispersed material permit the study of the interaction of the reinforcements in and with the liquid matrix, of significance in both processing phases, and the verification of composite product properties. They can, further, be scheduled early since extensive directly-applicable information is available from contract NAS8-27806 "Preparation of Composite Materials in Space."

Experiments with fiber/particle composites are, therefore, divided into two groups:

- (1) Experiments with pre-dispersed material
- (2) Experiments with zero-g mixing.

The first group is discussed in this section, and the second group in Section 7.4.

#### 7.3.1 Process Definition and Objectives

The preparation of composites from pre-dispersed material is primarily an experimental process for the purpose of obtaining data on the characteristics and properties of zero-g produced composites with a minimum of equipment and methods development. It may, however, also find application as a practical space manufacturing process. It consists essentially of (1) the preparation of the pre-dispersed ingot and (2) the joining of the component materials into a composite material by means of a zero-g matrix melting and solidification cycle.

The pre-dispersed ingot is prepared by solid-state ("dry") mixing and compaction of granulated matrix material and reinforcements. For low-g experiments the material is encapsulated by compaction directly into the sample container. Zero-g processing consists of heating to somewhat above the melting temperature of the matrix, temperature hold until all matrix material is molten, and cooling through solidification. Process evaluation consists of sample analysis after recovery and correlation with original sample composition and with temperature measurements during low-g processing.

### 7.3.2 Verification Requirements

In earlier studies of fiber/particle composites (Contract NAS8-27806 "Preparation of Composite Materials in Space") it was found that the complexity of the composite preparation depends primarily on the processing temperature. As evidenced in Fig. 7.3-1 there is a wide choice of reinforcements compatible with matrix materials of lower melting temperature, such as aluminum. For composites of higher processing (matrix melting) temperature, considerable developmental work is required to define compatible and effective matrix-reinforcement combinations.

Another criterion for process complexity is the casting mode, whether the composite is processed in the original mold, or prepared in a supply container and transferred into a mold or molds. These criteria indicate the division of experiments into three groups or verification levels:

- Level I: Processing of composites with a matrix melting temperature below 1000° C in the original mold.
- Level II: Processing of composites with matrix melting temperatures above 1000° C in the original mold.
- Level III: Casting of composites into molds from a melting and supply chamber.

For this program, a confinement to Verification Level I is indicated since it minimizes the number of variables - much of them related to material problems, rather than processing problems - yet, at the same time, generates the data required to move to higher verification levels.

In the cited study it was further found that the preparation techniques for the component materials, such as treatment for wettability, have to be developed specifically for each individual matrix metal; it was, therefore, recommended to limit initial low-g processing experiments to one matrix material with a variety of reinforcements. Aluminum was selected as the logical choice since it combines the stated requirements and limitations with high practical usefulness.

The choice of aluminum as base material for all composite experiments places the processing temperature at 700° C. Level I verification calls, therefore, for the capability of heating to 700° C and solidification within the available low-g time. It further calls for the following measurements:

- (1) Pre-Processing: Accurate material composition
- (2) Low-g Processing: Material temperature and g-level vs time.
- (3) Post-Processing: (a) Analysis of microstructure with regard to reinforcement distribution; (b) analysis of the micro-structure with regard to metallurgical characteristics; (c) mechanical properties of the composite, such as strength; (d) correlation of (a) (b) (c) with measurements (1) and (2), above.

### 7.3.3 Experimental Materials

The composite material is defined by three values: (1) the matrix material (2) the reinforcement material and (3) the reinforcement content.

(1) Matrix Material: The selection of aluminum as the sole base material still leaves a wide choice of specific Al-alloys. The alloy selection is primarily governed by wetting characteristics and mechanical properties. Most promising are Al alloys with 5% Cu for improved wetting characteristics and less than 1% Si for high fluidity. Such alloys also exhibit high strength and, with the addition of Mg, high response to strengthening heat treatment after composite processing. The alloy properties of interest for this evaluation are:

Density ( $\rho$ ) (gr/cm <sup>3</sup> )	2.69-2.80	Liquidus Temperature (° C)	616-660
Liquid-State Viscosity ( $\mu$ ) (centipoise)	1.1-1.2	Solidus Temperature (° C)	500-643
Min. Strength - as cast (psi)	35,000		
Typical Strength - heat treated (psi)	55,000		

(2) Reinforcement Materials: The following reinforcement materials have been tentatively selected (in the order of preference):

- (a) Graphite Fibers
- (b)  $Al_2O_3$  Fibers
- (c)  $Al_2O_3$  Whiskers
- (d) Mixture of (b) and (c) with  $Al_2O_3$  Particles
- (e) Si C Whiskers
- (f)  $Al_2O_3$  Particles Only.

All reinforcement materials exhibit low wetting characteristics with regard to Al (somewhat improved for the defined alloys) and have to be treated for wettability. All fiber materials (a) (b) will further be pre-coated with the matrix alloy.

(3) Reinforcement Content. The maximum possible reinforcement content is limited by the geometry of the reinforcement framework and is highly dependent upon the reinforcement L/D. The max. possible contents as related to L/D are identified in Fig. 7.3-2. It shows, that it ranges from 60% for particles to 14% for high L/D fibers. The strengthening effect is, however, not solely determined by fiber content; earlier studies showed, that for whisker-particle mixtures, substantial strengthening effects are obtained with contents as low as 0.1%. Since the limited number of experiments precludes wide content variations, the following contents (by volume %) have been tentatively selected:

Fibers (L/D 20-50): 4% and 12%

Whiskers (L/D 50-200): 4% and 8%

Whiskers and Particles: 0.1% and 5%

Particles Only: 1%

The absolute length of whiskers ranges from 1 to 4 mm, with an average max. length of 3 mm. This value has also been selected as max. fiber length (which can be chosen).

The exact contents are formulated as part of the "dry" mixing process prior to sample compaction.

#### 7.3.4 Material Quantity and Sample Size

The required minimum amount of experimental material is determined by two criteria:

- (1) According to the results of the cited prior study, the ratio of container diameter to reinforcement length ( $C/L$ ) has to be at least = 4 to minimize the disturbance of the reinforcement distribution along the container wall. Since the max fiber length is in the order of 3 mm, the sample has to be at least 1.2 cm in diameter.
- (2) The min. sample length is dictated by length required for strength (tensile, creep) tests which is in the order of 7 cm.

A standard sample size of 1.2 diam. x 7 cm has, therefore, been adopted. For shorter reinforcements and particles the use of a flat, rectangular sample may be considered which would be easier to compact and permit the preparation of several tensile specimen from the same heat (experiment). The major data of the two sample types are as follows:

Configuration	Cylindrical	Flat	
Dimensions	1.2 dia x 7	6 x 3 x 0.8	cm
Volume	7.9	9.6	cm <sup>3</sup>
Weight (app.) <sup>*)</sup>	22	27	gr
Heat Content (20-700° C)	5,200	6,400	cal

\*) Depending on reinforcement type and content.

Sample Container. Fiber and particle composites are exclusively processed in a mold, since this is representative of practical applications (composite casting). The sample container has to match the thermal expansion of the sample material, either by design or material. The container walls have to be wetted by Al to assure contact and heat transfer. Unfortunately, most of the materials wetted by Al are also soluble in Al, such as Cu. Container materials and coatings to improve wetting characteristics are presently evaluated under another study.

### 7.3.5 Experimental Process Definition

The preparation, performance and evaluation of low-g experiments with pre-dispersed composites comprises the following operational phases:

- 1) Preparation of the component materials, dosaging for exact composition, dry mixing and compaction into the sample container. During all these operations, carried out in the laboratory, the materials have to be kept under uninterrupted protection against oxidation by a high-purity argon atmosphere. Preparation of the materials includes the preparation of the matrix powder and surface treatments of both, matrix and reinforcements.
- 2) Installation of the (sealed) sample capsule in the experiment apparatus and the low-g test facility.
- 3) Low-g processing, consisting of heating through matrix-melting to the processing temperature of 700° C. hold at this temperature for a pre-determined period, followed by induced cooling through solidification. Measurements during this melt cycle are sample temperature and g-loads, both vs. processing time.
- 4) Sample recovery and removal from container.
- 5) Sample evaluation, consisting of photomicrographic analysis, strength tests and correlation of results with the sample composition (1) and the in-process measurements (3).

The sequence of the individual operational steps is identified in the Process Flow Diagram, Fig. 7.3-3. (Bold frames indicate g-sensitive process phases; dotted frames indicate optional phases).

### 7.3.6 Low-g Test Requirements

As indicated in the flow diagram, Fig. 7.3-3, the g-sensitive time extends from a temperature below the melting point of the matrix through liquid-state processing to completed solidification. The rate of heating and cooling have no processing significance and may be as high as possible. The "hold" time at processing temperature (after complete



sample melting) should be at least 20 seconds; the shape of the temperature profile during this period is unnessential as long as it remains above the melting temperature. The required total low-g processing time depends on the methods of heating and cooling. The two most adaptable heating methods which may be used alternately, are electrical resistance elements or exothermic heating. In both cases, the heat transfer to the sample is by radiation. The following evaluation is based on the use of modular processing units.

**7.3.6.1 Time Requirements - Electrical Heating.** The electrical furnace for standard 1.2 x 7 cm samples is described in 6.2.1 and illustrated in Fig. 6-4. For the heating element temperature of 1250° C and a contained sample, no argon atmosphere is required. Terminal cooling is accomplished with a closed system, also shown in Fig. 6-4.

The heating of a cold furnace requires an initial power of 1800 watts. The concurrent heating of several units would result in an excessive power peak and ground-preheating to 550° C is mandatory. The resulting sample temperature profile is shown in Fig. 7.3-4. The power and processing time data are as follows:

	<u>Max Watts</u>	<u>Time/Sec</u>
Ground Pre-heat	1800	60
Ground Hold	400	to 1800
Flight Hold	400	= or > 60
Flight Heating to 700° C	800	90*
Flight Hold at 700° C	500	20*
Flight Solidification	-	20*
Flight-Total Power (Max)	41 Wh	
Flight-Total Low-g (*) Time		130 Sec

**7.3.6.2 Time Requirements - Exothermic Heating.** The exothermic module including a terminal cooling system to be used for the standard 1.2 x 7 cm sample is described in Sec. 6.2.2 and illustrated in Fig. 6-8. The temperature profile of the sample is shown

in Fig. 6-9c. The thermal gradient in the sample will necessarily be much higher than by electric heating and the material close to the mold wall may reach a temperature of 1100° C which is, however, acceptable. Since there are no power considerations involved, all exothermic units can be operated concurrently. In the absence of experimental data, the time required for complete sample melting cannot be exactly defined; it is estimated to be in the order of 30-60 seconds. The sample will then remain in the liquid state for at least 60 seconds due to the high amount of heat stored in the unit. According to the data defined in 6.3.3, sample solidification by active terminal cooling is achieved in app. 40 seconds. On the basis of conservative assumptions, the min. total low-g time is computed as follows:

Heating	60 sec
Liquid state hold	30 sec
Solidification	40 sec
Min. low-g time	130 sec

If longer low-g test times are available, it will be expedient to extend the liquid-state hold time accordingly.

7.3.6.3 g-Level. During the total low-g periods defined above, any g-forces acting on the sample should not exceed  $10^{-3}$  g.

### 7.3.7 Low-g Facilities and Experiments

The identified low-g time of 130 sec places the experiments in the typical rocket regime. The number of experiments which can be carried out on one flight is determined by payload weight, space and time limitations. The following evaluation is based on rocket class 1 and trajectory A (Standard WSMR capability).

#### 7.3.7.1 Payload Weight Limitations (Equipment data see Sect. 7.3.8).

	<u>Weights (kg)</u>	<u>(kg)</u>
<u>Rocket Payload Capability</u>	130	

#### Electrical Furnaces

Support Module	57.0		
5 Proc. Modules	67.5	124.5	5.5

#### Exothermic Furnaces

Support Module	45.0		
5 Proc. Modules	65.0	110.0	20
6 Proc. Modules	78.0	123	7

#### 7.3.7.2 Payload Space Limitations (Measured in axial height available in payload can)

	<u>Electrical Furnaces</u>	<u>Exothermic Furnaces</u>
Available Space	80 cm	100
5 Proc. Modules	80 cm	95
Margin	0 cm	5

7.3.7.3 Experiment Time Limitations- Electrical Furnaces. While the low-g time of the individual experiment of 130 sec is well within the available time of 243 seconds, the number of experiments is limited by power limitations. However, the 5 experiments feasible from weight and space considerations can be accommodated by the following scheduling of experiments (numbers in chart identify processing modules 1 to 5).

<u>Low-g Time (Sec)</u>	<u>Hold/550° C (x Sec) (400w)</u>	<u>Melting (90 Sec) (800w)</u>	<u>Hold/700° C (20 Sec) (500w)</u>	<u>Solidific. (20 Sec) -0-w)</u>	<u>Total Power (Watts)</u>
0-90	3, 4, 5	1, 2	-	-	2, 800
90-110	4, 5	3	1, 2	-	2, 600
110-180	-	3, 4, 5	-	1, 2	2, 400
180-200	-	4, 5	3	-	2, 100
200-220	-	-	4, 5	3	1, 000
220-240	-	-	-	4, 5	-0-

7.3.7.4 Experiment Time Limitations - Exothermic Furnaces. The exothermic modules could be started concurrently upon reaching low-g (low-g time 0). However, the margin between the experiment time (130 sec) and the available time (243 sec) permits staggering which is desirable to minimize the steam exhaust peak during initial cooling. Start of the five experiments at 20 sec intervals results in a total low-g time of 210 seconds, leaving a safety margin of 33 seconds.

7.3.7.5 Experiment Definition It is concluded on the basis of the foregoing evaluations, that predispersed composite experiments can be effectively carried out on research rocket class 1 and trajectory A, either with electrical radiation furnaces, or with exothermic furnaces. A total of 5 experiments can be accommodated per flight for either heating method. The total low-g time required is well within the available time of 243 seconds, as evidenced by the following data:

Low-g Time Capability	
RR-1, Traj. A	243 sec
5 Experiments, Electric Furnaces	240 sec
5 Experiments, Exothermic Furnaces	210 sec

#### 7.3.8 Apparatus and Payload Definition

The payload consists of the support module and five (5) processing modules representing the apparatus. No other equipment is required. The use of contained samples and the limitation of the electrical heating elements to 1250° C eliminates the need for atmosphere control in the electrical furnaces. In the exothermic furnaces, chamber wall oxidation is minimized by a thin platinum coating of the Ta (or Nb) wall.

The payload assessment is based on the use of the standard cylindrical 1.2 x 7 cm sample. The payload requirements for the alternate use of the flat 6 x 3 x 8 cm sample is essentially the same (electrical furnace only). There is a choice of two processing units:

- (1) Electrical Radiation Furnace
- (2) Exothermic Furnace

7.3.8.1 Electrical Processing Module. The applicable module, using electrical heating elements at 1250° C and terminal cooling with a closed system (water) is defined in Sect. 6.2.1.2 and Fig. 6-4. Its major physical and functional data are:

Dimensions	24 diam x 16 cm high
Weight	13.5 kg
Preheating to 550° C	1,800 w for 40 sec
Hold at 550° C	400 w (to 1 hour)
Melting (550-700° C)	800w for 90 sec
Temp hold at 700° C	500w for 20 sec
Solidification	-0- for 20 sec
Final cooling	-0- for 40 sec.

7.3.8.2 Exothermic Processing Module. The exothermic heating unit is defined in Sect. 6.2.2. ("Size II") and the module assembly including cooling system illustrated in Fig. 6-8. Its major data are:

Dimensions	30 x 16 x 19 cm high
Weight (incl. coolant)	13.0 kg
Heating/melting time	60 sec
Liquid-state temp hold	30 sec
Solidification	40 sec
Final cooling	240 sec

Detailed cooling data are listed in Sect. 6.3.3.

7.3.8.3 Support Module. The support module for the electrical processing module requires substantial power supply and conditioning equipment (2 batteries, inverter, 2.5 kw transformer). For exothermic processing, only 1 battery and a simple 28V-DC power control and distribution unit is needed; it requires, however, a central argon supply unit for atmosphere control in the processing chamber to prevent oxidation of the chamber wall (Ta). The outfitting, weight and space requirements of the two versions of the support module are as follows (numbers in parentheses identify components specified in Sect. 5.2.2 and Table 5-1):

#### Support Module - Electrical Furnaces

Basic Structure (1, 2, 3)	33	kg
2 Batteries (4)	6	kg
Power Conditioning (5-A, B, C-2)	12	kg
Sequencer, Recorder (6, 7)	4	kg
Contingency (8)	2	kg
<hr/>		
Total Weight	57	kg

Net Space Available for Processing Modules (axial height)	80	cm
--------------------------------------------------------------	----	----

#### Support Module - Exothermic Furnaces

Basic Structure	33	kg
1 Battery	3	kg
Power Distribution/Controls (5A)	3	kg
Sequencer, Recorder	4	kg
Contingency	2	kg
<hr/>		
Total Weight	45	kg

Net Space for Processing Modules (axial height)	100	cm
----------------------------------------------------	-----	----

7.3.8.4 Payload Assembly. The payload assemblies for predispersed composite experiments are shown in Fig. 7.3-5 (electrical furnaces) and 7.3-6 (exothermic furnaces). The apparatus in either version consists of 5 processing modules. The total payload weights are as follows:

### Electrical Furnaces

5 Proc. Modules (13.5 kg)	67.5	kg
Support Module	57.0	kg
Total Payload Weight	124.5	kg
Rocket 1-A Capability	130.0	kg
Reserve	5.5	kg

### Exothermic Furnace

5 Proc. Modules (13.0 kg)	65	kg
Support Module	45	kg
Total Payload Weight	110	kg
Rocket 1-A Capability	130	kg
Reserve	20	kg

## 7.3.9 Experiment Performance

7.3.9.1 Ground Operations. Operations at the launch site prior to count-down consist of:

- (1) Dry experiment assembly check-out
- (2) Charging with expendables (water, gas)
- (3) Second check-out (functional, measuring)
- (4) Vehicle installation
- (5) Final check-out.

Operations during count-down:

- (6) Pre-heating (not applicable to exothermic payload)

7.3.9.2 Flight Operations. The sequencing of flight operations are pre-set and are defined in the time diagram, Fig. 7.3-7 for electrical heating and 7.3-8 for exothermic heating.

7.3.9.3 Post-flight Operations at the launch site consist of:

- (1) Payload Recovery

- (2) Payload Removal from Vehicle Section
- (3) Sample Recovery for Evaluation
- (4) Recovery of Recorder Tape and Telemetry Records for Evaluation
- (5) Cleaning of Payload Assembly



Reinforcements	Matrices						
	Al	Mg	Fe	Ni	Co	Cr	Ti
SiC	●	●	C	C	C	C	C
Si <sub>3</sub> N <sub>4</sub>	●	●	C	C	C	C	C
Al <sub>2</sub> O <sub>3</sub>	●	●	●	●	●	C	C
C	●	●	C	C	C	C	C
BN	●	●	X	X	X	X	X
B	●	●	X	X	X	X	X

- Compatible, yet nonwetting; coating required.
- C Not compatible due to chemical reactivity.
- X Not compatible; reinforcement degradation due to temperature.

Figure 7.3-1. Compatibility of Reinforcements With Liquid Matrices

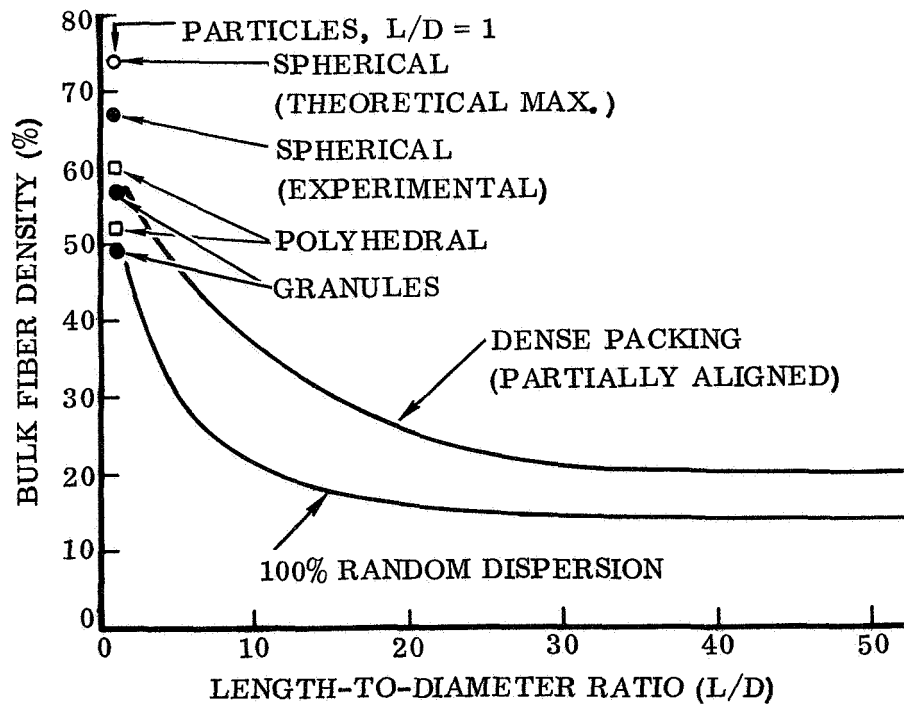


Figure 7.3-2. Maximum Volume Fraction of Random Dispersed Fibers and Particles (semi-infinite container,  $C/L \approx \infty$ )

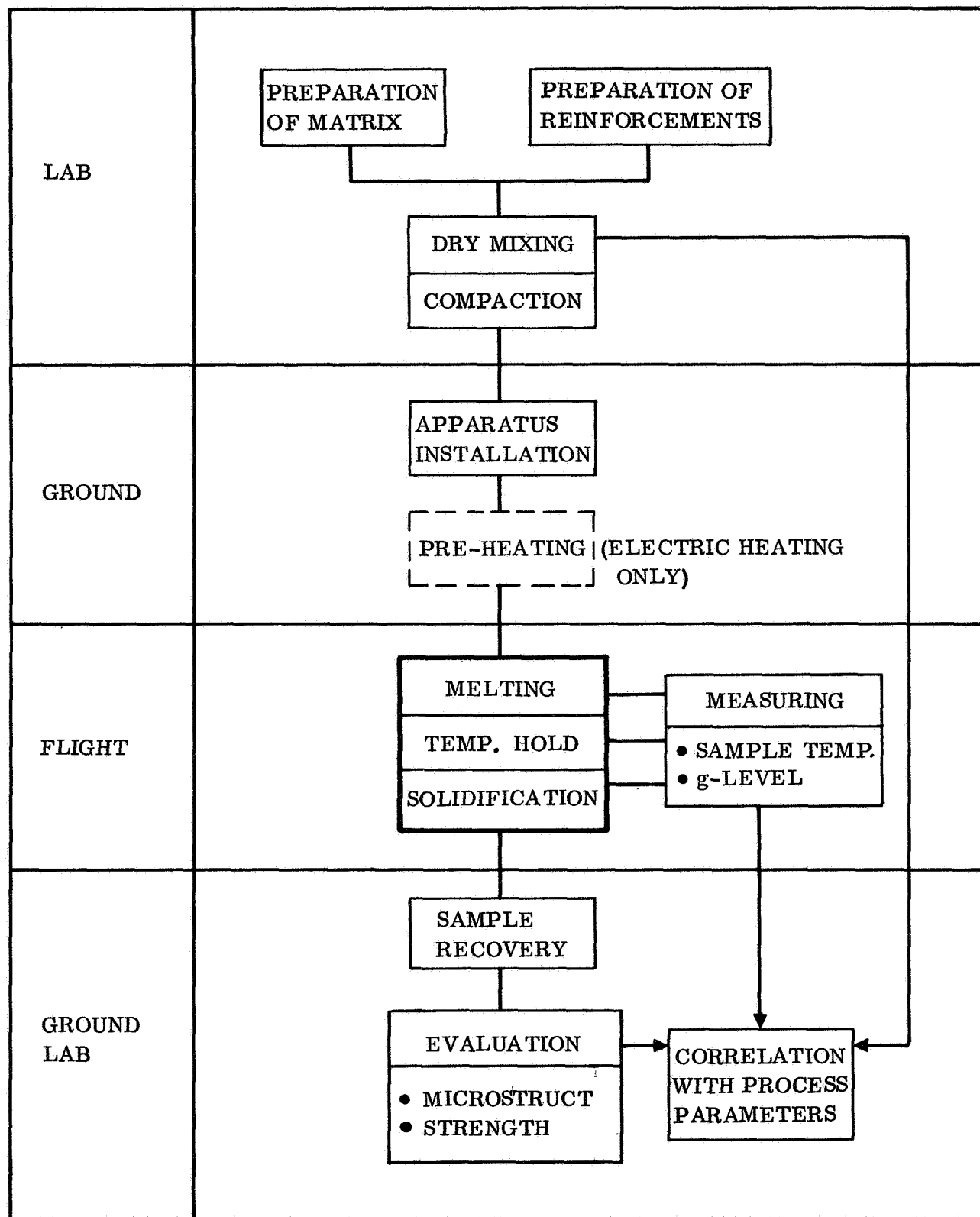


Figure 7.3-3. Process Flow Diagram — Predispersed Fiber/Particle Composites

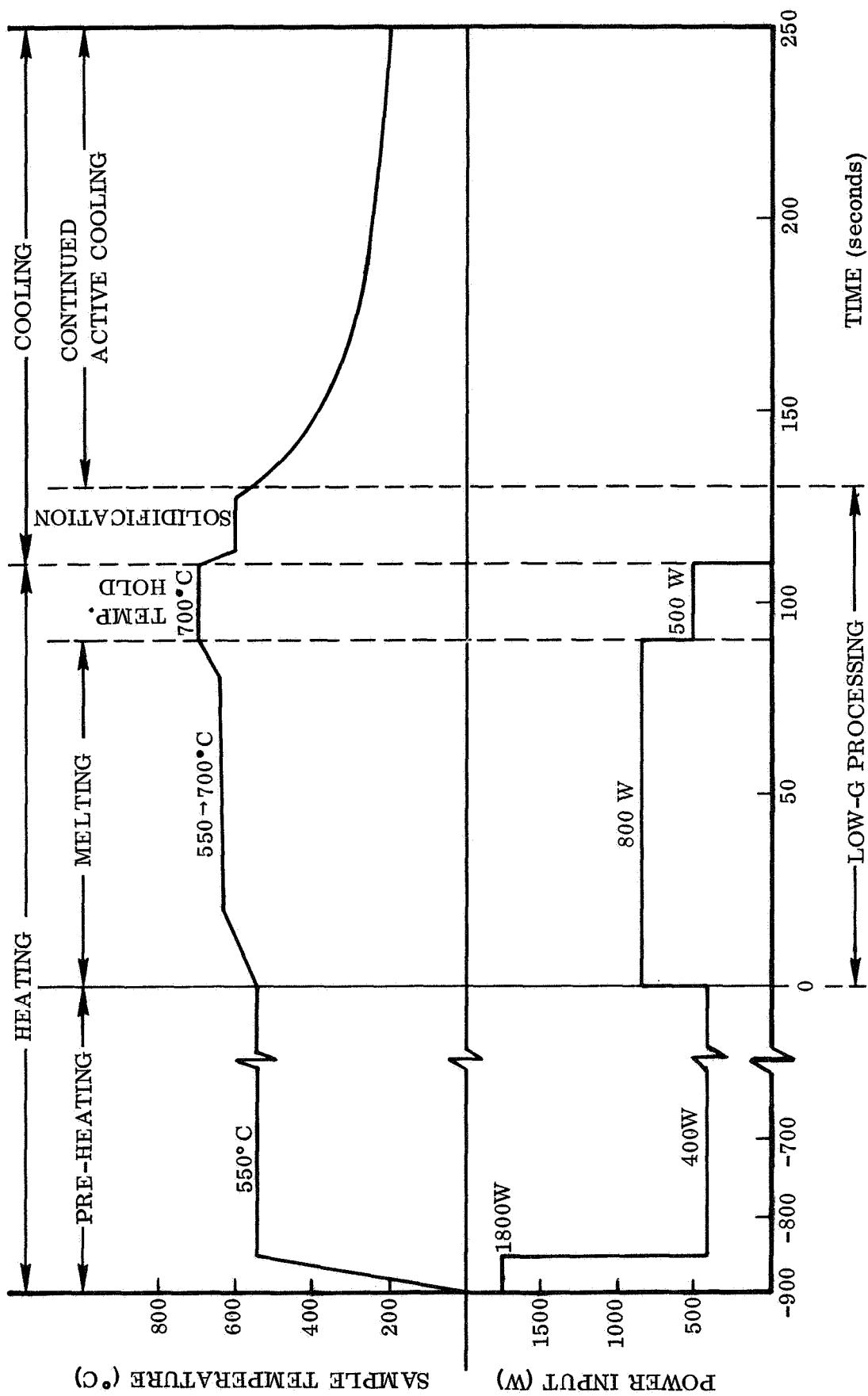


Figure 7.3-4. Sample Temperature and Power Profiles -- Processing of Composites in Electrical Furnaces

# ROCKET PAYLOAD ASSEMBLY

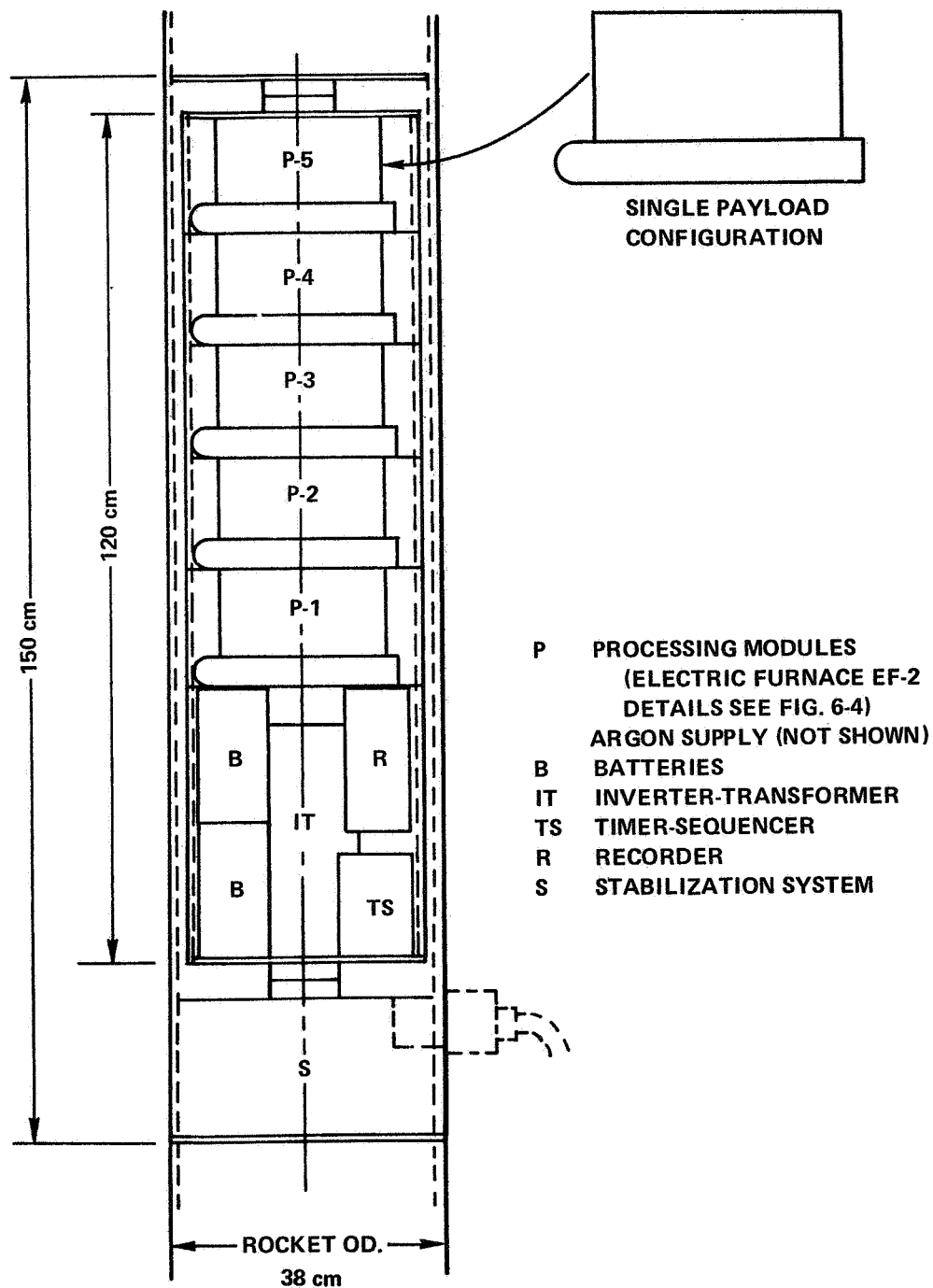


Figure 7.3-5. Payload Assembly — Fiber/Particle Composites (Predispersed) — Electric Furnaces

## ROCKET PAYLOAD ASSEMBLY

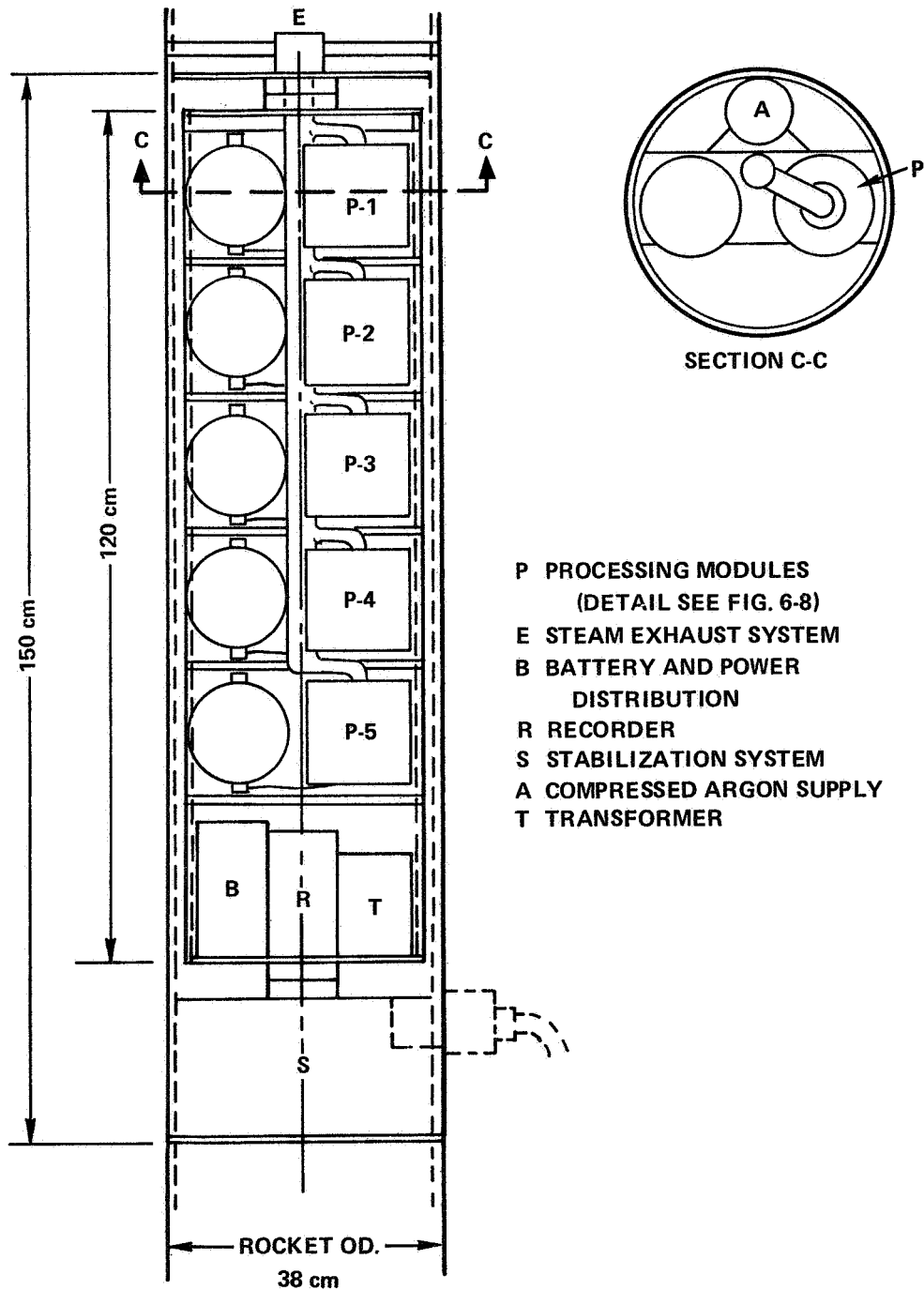


Figure 7.3-6. Payload Assembly — Fiber/Particle Composites  
(Predispersed) — Exothermic Furnaces

# EXPERIMENT PROGRAM - TRAJECTORY A

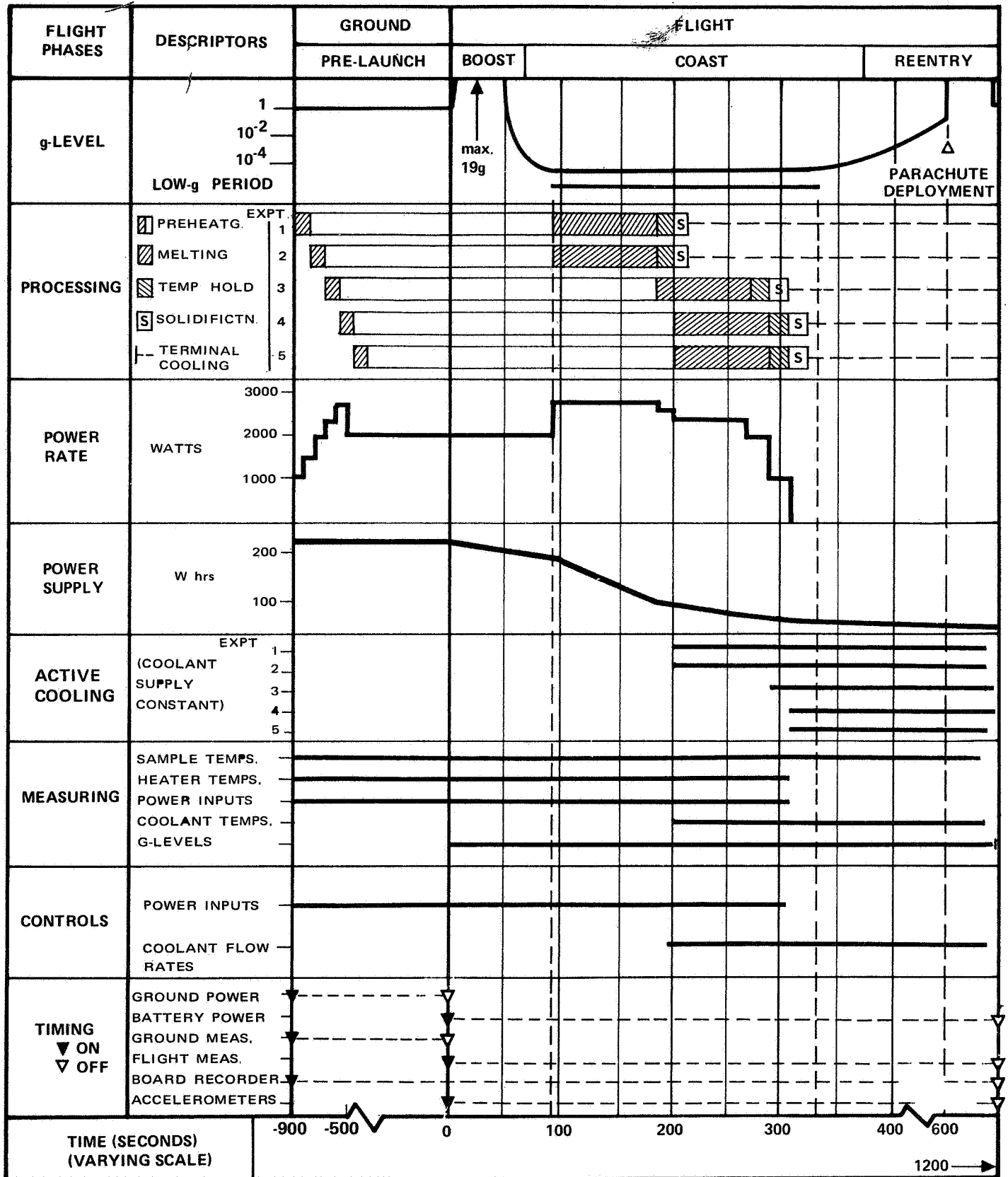


Figure 7.3-7. Time Diagram - Predispersed Composites - El. Heating

# EXPERIMENT PROGRAM – TRAJECTORY A

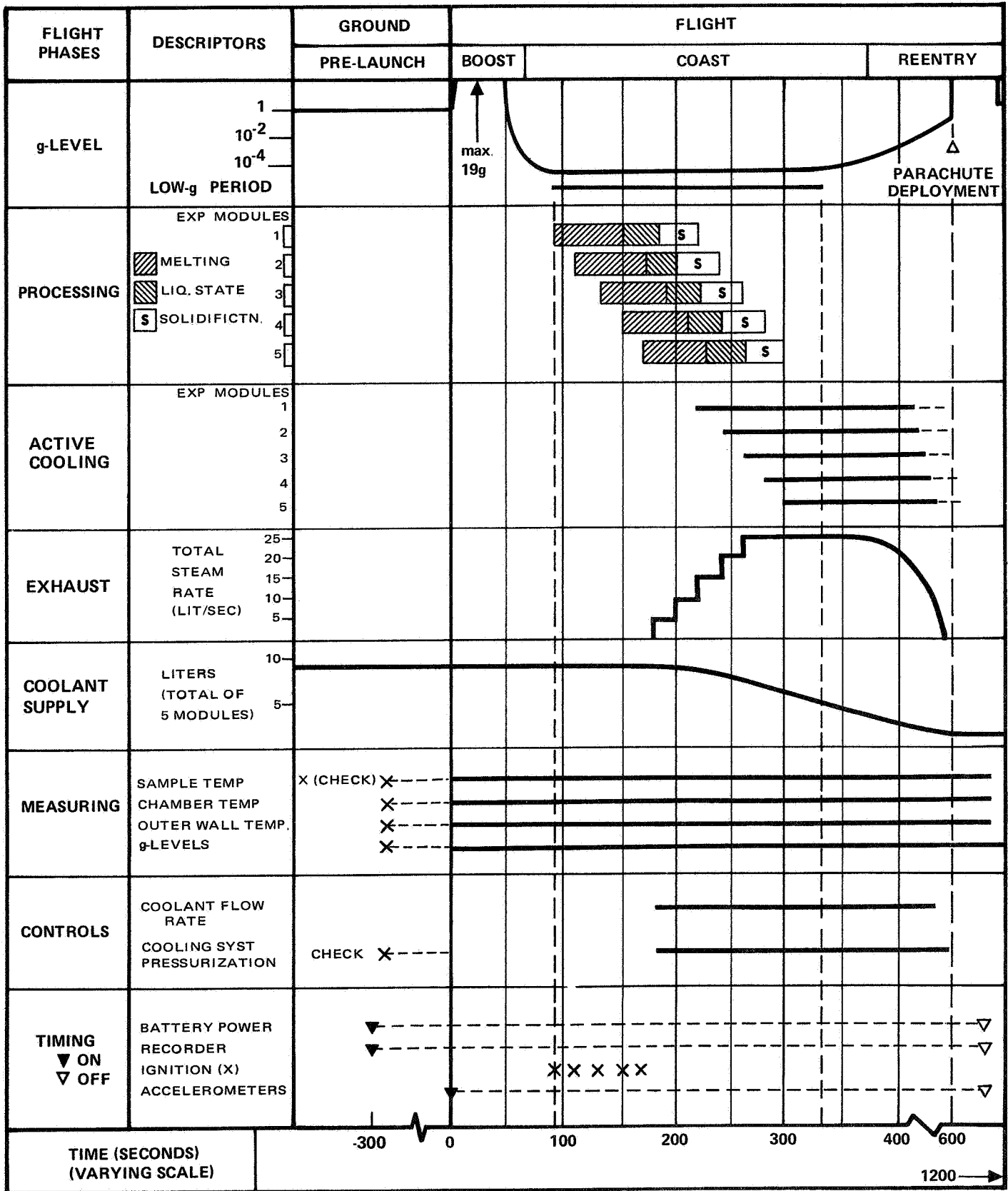


Figure 7.3-8. Time Diagram - Predispersed Composites - Exothermic Heating

## 7.4 FIBER/PARTICLE COMPOSITES - LOW-G MIXING

### 7.4.1 Process Definition and Objectives

The basic criteria for the preparation of composites by liquid-state processing and the resulting product characteristics are established in experiment 7.3. The objective of this experiment is to generate the dispersion of reinforcements by liquid-state mixing under low-g conditions. If successful, it represents a practical process, adaptable to large-scale production of composites materials and finished components in space. The specific objectives of low-g experiments are

- (1) To investigate the dispersion characteristics attainable by dynamic mixing
- (2) To investigate the effectiveness of various mixing modes and techniques
- (3) To obtain data on the product characteristics and properties.

The experimental process consists of 1) preparation of a pre-material, containing the reinforcements in segregated position, as it occurs naturally in one-g mixing of the reinforcements with a molten matrix, 2) re-melting under low-g conditions and dynamic mixing, 3) solidification of the resulting mixture.

### 7.4.2 Verification Requirements

The requirements for the verification of the characteristics implied by the stated objectives are:

- (1) Preparation of sample materials with varied combination of
  - (a) Matrix composition
  - (b) Reinforcement material type and content
- (2) Low-g processing varying
  - (a) Mixing technique
  - (b) Mixing mode
- (3) Facilities for sample processing (2)
- (4) In-process measurements of material temperature and g-levels vs. time



(5) Sample recovery and evaluation for

- (a) Effectiveness of mixing technique and mixing mode as to the dispersion of reinforcements
- (b) Metallurgical effects of low-g processing and correlation with component material characteristics
- (c) Mechanical properties of the obtained composite material and correlation with (a) and (b), above, and with (4).
- (d) Reconstruction of the fluid mechanics of the material during the mixing process.

Based on the considerations outlined in 7.3.2, three verification levels with increasing experiment complexity have been defined:

Level I: Single base material with moderately high melting temperature and one mixing technique.

Level II: Introduction of additional mixing techniques, material as in I.

Level III: Application of the most effective mixing technique evolving from I and II to other base materials, including high-temperature materials.

To minimize the number of variables, initial experiments and the following experiment definition have been limited to verification level I. In conformance with the experiments of 7.3, aluminum (and alloys) has been selected as base material. The selected mixing technique is ultrasonic agitation; it serves as a model, since the resulting experiment envelope (number of experiments per test, equipment weight and dimensions etc) will be essentially the same for other mixing techniques.

#### 7.4.3 Experiment Materials

Experimental materials are identical to those selected for experiments 7.3 and defined in Section 7.3.3, except for the reference to compaction which, in this case, is replaced by casting of the pre-material.

#### 7.4.4 Material Quantity and Sample Configuration

(As defined in Section 7.3.4)

#### 7.4.5 Experimental Process Definition

The preparation, performance and evaluation of low-g experiments comprises the following major phases:

- (1) Preparation of the component materials, dosaging for exact composition and casting into the sample container.
- (2) Installation of the sample in the experiment apparatus and the low-g test facility.
- (3) Low-g processing consisting of:
  - I. Melting and heating to 700° C
  - II. Liquid-state mixing, including stilling (deceleration of mixture)
  - III. Cooling thru solidification (700 ~ 450° C)
  - IV. Terminal cooling
- (4) Sample recovery and removal from container
- (5) Sample evaluation, consisting of photomicrographic analysis, strength tests and correlation of results with sample composition and processing parameters measured during Phase (3).

The sequence of individual operational steps is identified in the Process Flow Diagram, Fig. 7.4-1 (Bold frames indicate g-sensitive process phases).

#### 7.4.6 Low-g Test Requirements

In this experiment, only Phases II and III, as identified above, are g-sensitive; in contrast to other composite experiments, the distribution of the mixture during melting is immaterial and Phase I can be carried prior to the low-g period. This permits a (desirable) extension of the stilling period of Phase II (deceleration of the agitated mixture).

**7.4.6.1 Time Requirements.** The processing time and power requirements for the defined sample material and quantity are:

Processing Phase	x-g Time (Sec)	Low-g Time (Sec)	Max. Power (Watts)
I. Melting	150		1800
Hold at 700° C	0-600		500
II. Mixing/Stilling		80	600
III. Solidification		20	100
IV. Terminal Cooling	300-600		100
Total Low-g Time		100	
Total Low-g Energy			11 wh
Min. Total Time incl. Melting	230		

7.4.6.2 g-Level. During Phases II and III any forces acting upon the sample should not exceed  $10^{-4}$  g.

#### 7.4.7 Low-g Facilities and Experiments

The low-g time of 80 seconds places the experiments in the typical rocket regime. Since the most power- and time-consuming processing Phase I (melting) can be carried out on the ground prior to launch, the max. number of experiments per flight is determined by equipment limitations. A cursory review of the equipment data, Section 7.4.8, shows that the critical limitation is payload space.

##### 7.4.7.1 Payload Space Limitations (Measured by axial height in payload can).

Required Space - 4 Expt. Modules	80 cm
Available Apparatus Space - RR-1	80 cm
Surplus	- 0 -

##### 7.4.7.2 Payload Weight Limitations

Support Module	57 kg
4 Expt. Modules	60 kg

Ultrasonic Generator	2 kg
Total Weight	119 kg
RR1-A Payload Capacity	130 kg
Surplus	11 kg

7.4.7.3 Time and Power Requirements. The following assessment of experiment timing and the resulting power requirements is based on flight time, since the power levels for the temperature hold (after ground melting) during the boost phase are significant. Low-g time extends from 90 - 333 flight seconds for the selected trajectory A. The numbers in the chart denote experiments 1 to 4.

<u>Flight Time</u> <u>(Sec)</u>	<u>Hold at 700° C</u> <u>(500W)</u>	<u>Mixing</u> <u>(600 W)</u>	<u>Solidif.</u> <u>(100W)</u>	<u>Term. Cooling.</u> <u>(100W)</u>	<u>Max. Power</u> <u>(W)</u>
0-90	1, 2, 3, 4				2, 000
90-170	3, 4	1, 2			2, 200
170-190	3, 4		1, 2		1, 200
190-270		3, 4		1, 2	1, 400
270-290			3, 4	1, 2	400
290-670				1, 2, 3, 4	400

Power does not exceed the max. support module of 3,100W (2,800 transformed to AC).

The total power consumption is 197 wh including support equipment (recorder etc), which is within the total power supply of 220 wh.

7.4.7.4 Experiment Definition. The results of this assessment are summarized as follows:

Low-g Facility	RR Class 1
Trajectory	A (Standard WSMR)
Expts. per Flight	4
Total Low-g Processing Time	200 Sec
Max. Discharge Rate	2,400 W
Total Power Consumption	195 Wh

#### 7.4.8 Apparatus and Payload Definition.

The payload consists of the support module and the apparatus for four (4) experiments.

7.4.8.1 Apparatus. The apparatus consists of four (4) processing modules and a central ultrasonic energy generator.

The processing modules use electrical radiation heating and active circulation cooling (closed system). The configuration of the individual modules is illustrated in Fig. 6-4 and described in Sections 6.2.1.2 (furnace) and 6.3.2.2 (cooling system). Attached at the top of the processing chamber is the ultrasonic transducer measuring 5 x 5 x 4 cm. The acoustic energy is transferred to the sample with a "horn," reaching into the processing chamber and serving as sample support. The major data for the individual module and the 4-module assembly are:

	<u>1 Module</u>	<u>4 Module Ass'y</u>
Diameter	24 cm	24 cm
Axial Height	20 cm	80 cm
Weight (with coolant)	15 kg	60 kg
Power Rating (incl. pre-heat)	1800 w	3300
Flight Power Consumption	43 wh	170 wh
Coolant Supply (water)	2 liters	8 liters

The ultrasonic generator, serving all 4 experiments, is located in the support equipment section of the payload can. It is a solid-state device, receiving its power directly from the battery. Its major data are;

Dimensions	3 x 10 x 16 cm
Weight	1 kg
Weight of Leads etc.	1 kg
Total weight	2 kg

7.4.8.2 Support Module. In view of the substantial power requirements, the support module is equipped with 2 batteries and the larger transformer unit. The outfitting, weight and space requirements are (numbers in parentheses identify components specified in Section 5.2.2 and Table 5-1):

Basic Structure (1, 2, 3)	33 kg
2 Batteries (4)	6 kg
Power Conditioning (5-A, B, C-2)	12 kg
Sequencer, Recorder (6, 7)	4 kg
Contingency (8)	2 kg
	<hr/>
	57 kg

Net space available for processing modules	
(axial height)	80 cm

7.4.8.3 Payload Assembly. The payload assembly is shown in Fig. 7.4-2. The major payload data are as follows:

Payload Weight

Support Module	57 kg
4 Expt. Modules	60 kg
Ultrasonic Generator	2 kg
	<hr/>
Total Weight	119 kg

Power Supply

Total Power Supply	220 wh
Total Power Consumption	197 wh
Max Discharge Rate	2,400 w

7.4.9 Experiment Performance

7.4.9.1 Ground Operations. Operations at the launch site prior to launch consist of:

- (1) Dry expt. assembly check-out
- (2) Charging with expendables (water)
- (3) Second check out
- (4) Vehicle installation
- (5) Final check-out
- (6) Ground-activation of payload systems at -800 sec.
- (7) Preheating of each module to 700° C (sample melting) from ground power supply (1500 w = 200 sec) in the following sequence (in seconds prior to launch = time zero).

<u>Module</u>	<u>Melting (1800 w)</u>	<u>Hold (500w)</u>
1	-1000 to -800	-800 to 0
2	-800 to -600	-600 to 0
3	-600 to -400	-400 to 0
4	-400 to -200	-200 to 0

7.4.9.2 Flight Operations. The sequencing of flight operations is pre-set and is defined in the Time Diagram, Fig. 7.4-3.

7.4.9.3 Post-Flight Operations at the launch complex as defined in 7.3.9.3.

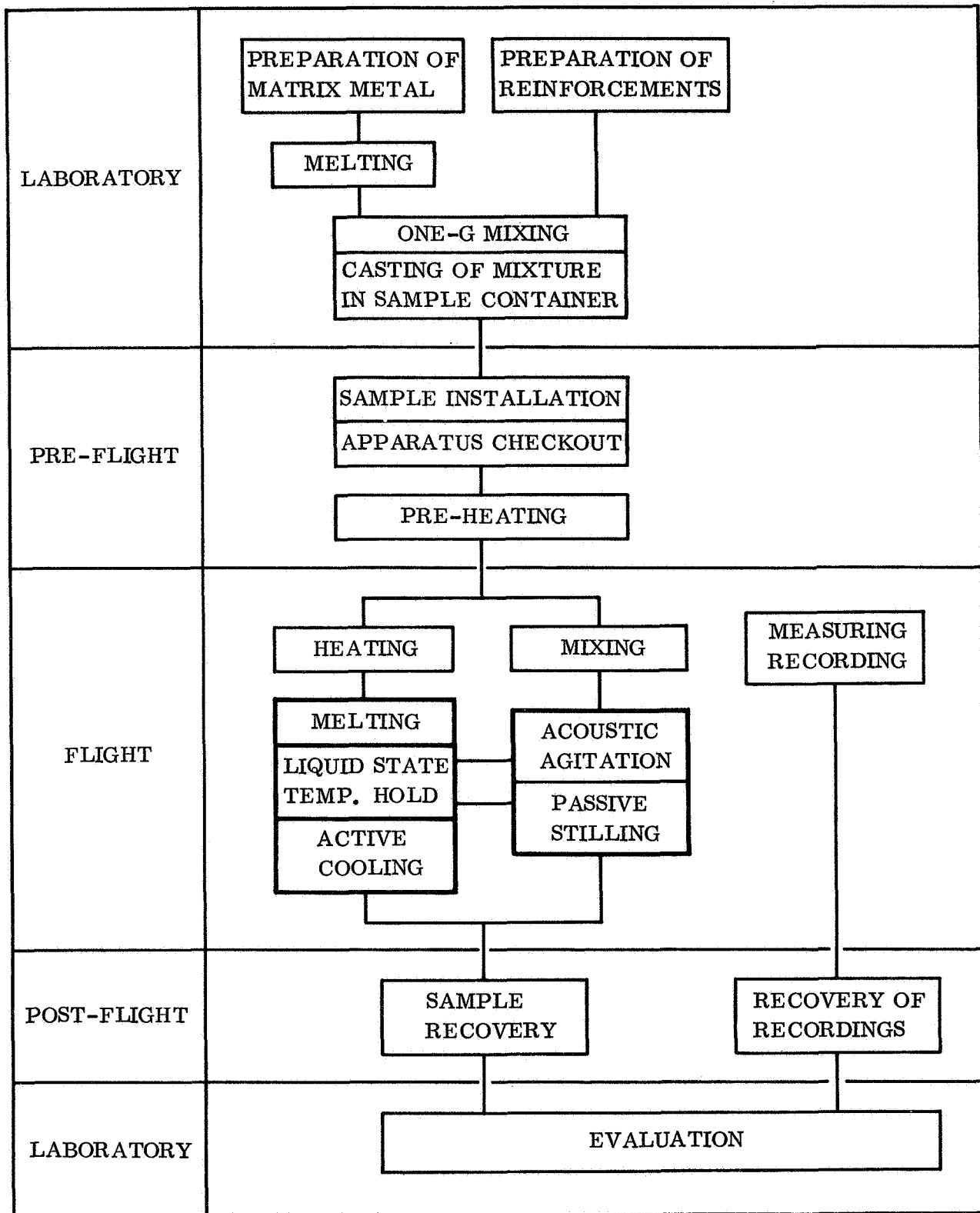


Figure 7.4-1. Process Flow Diagram - Fiber/Particle Composites - Low-g Mixing



# ROCKET PAYLOAD ASSEMBLY

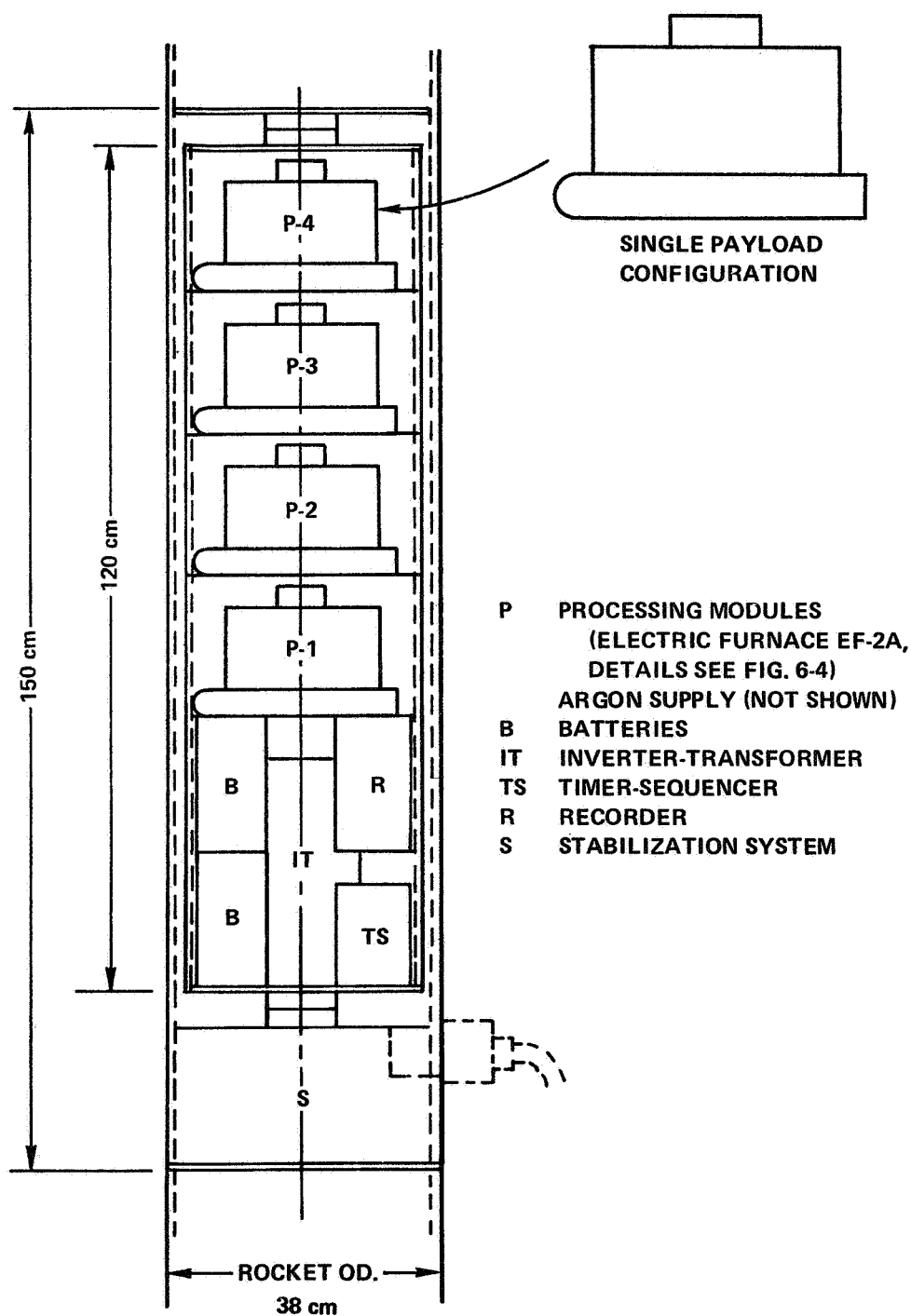


Figure 7.4-2. Payload Assembly — Fiber/Particle Composites — Low-g Mixing

# EXPERIMENT PROGRAM – TRAJECTORY A

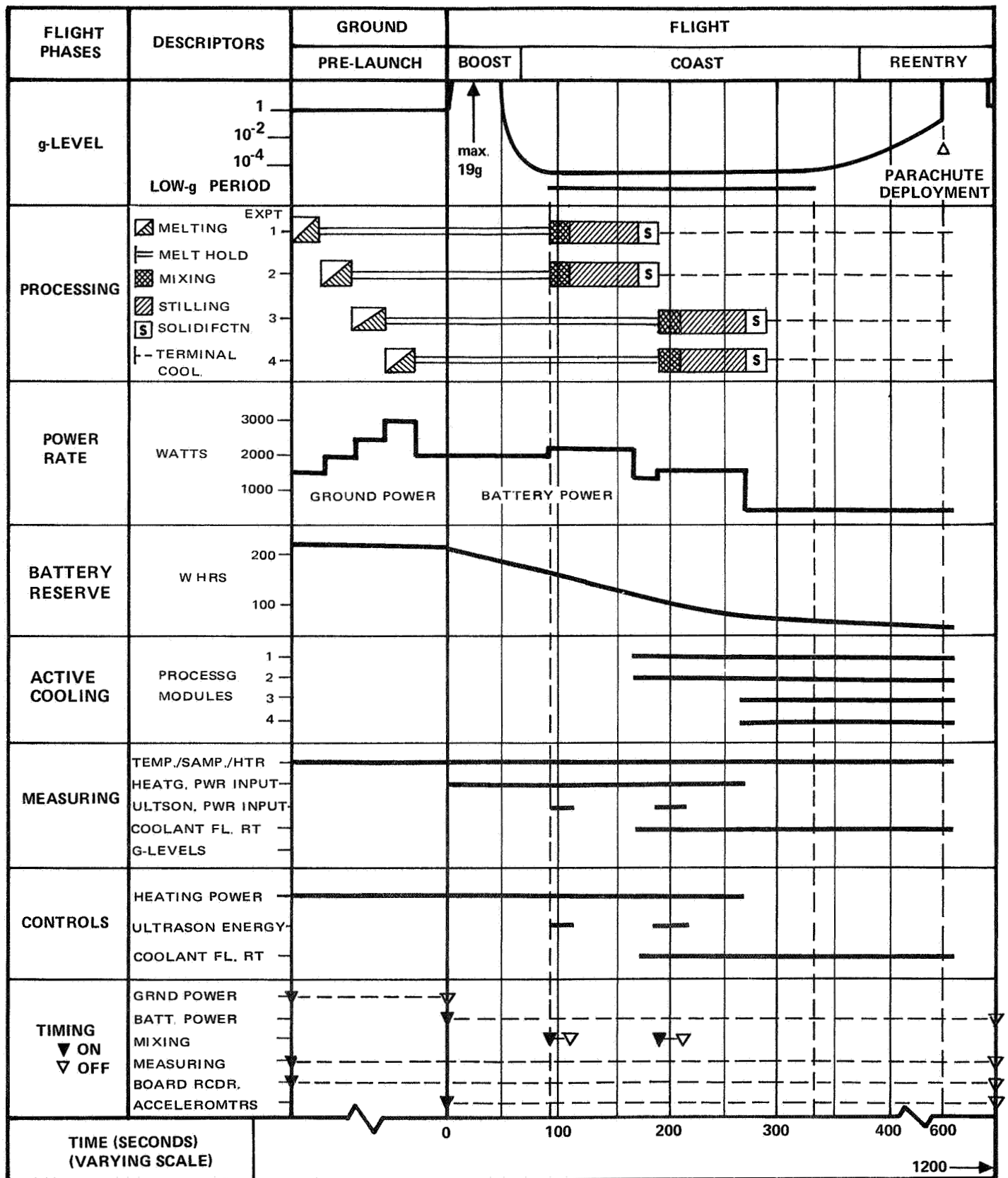


Figure 7.4-3. Time Diagram - Composites - Low-G Mixing (El. Heating)

## 7.5 CONTROLLED DENSITY METALS - PREDISPERSED COMPACT

This materials class comprises metals and alloys with reduced bulk density, achieved by dispersed gases. The gas dispersion can only be generated in the liquid (matrix) state; in view of the instantaneous segregation in one-g, a stable dispersion, which can be solidified into a product, requires a zero- or low-g environment. The preparation of controlled density metals (CDM) is, therefore, only feasible under low-g conditions.

Controlled density metals are also conveniently - but inaccurately - referred to as metal "foams." They comprise two types of products: 1) a composite metal and gas only (plain foams) with high ratio of stiffness to density, or merely low bulk density; 2) a composite of metal, reinforcements and gas (reinforced foams) with a high ratio of strength and stiffness to density.

Processes to produce CDM, both plain and reinforced, are distinguished by the mode of gas generation and dispersion (foaming method). The following methods have been defined conceptually (numbers refer to identification codes in Tables 1-3, Section 1.0).

- A2-1. Compact Foaming; the gas is generated by the decomposition of compounds which have been pre-dispersed in the solid state (particle mixture).
- A2-2. Gas Injection Foaming; in which the gas is added to the liquid metal in a continuous process.
- A2-3. Ultrasonic Foaming; in which a dissolved gas is driven out of the liquid metal by focused acoustic energy which, at the same time, induces agitation and dispersion.
- A2-4. Nucleate Foaming; in which a dissolved gas or matrix vapor is released from the molten metal by depressurization; the gas formation is induced, and the distribution controlled, by pre-dispersed solid nuclei (fine particles).
- 5. Cavitation Foaming; segregated liquid metal and gas are mixed by violent agitation (stirring or "beating").
- 6. Microsphere Foam; gas-filled microballoons are dispersed in a solid compact; conversion into a foam is achieved by a melt-cycle.

For initial experiments, process 1 is most promising and has been firmly adopted. Processes 3 and 4 have been eliminated earlier, since they call for additional research work. Process 2, selected earlier, has been tentatively deferred pending the results of drop tower experiments presently in preparation (Contract NAS 8-28056). In its place, method 5 has been selected, since it serves the same objective, yet is less complex and, therefore, more adaptable to initial experiments. Process 6, originally eliminated, has been reconsidered as an alternate as back-up for process 1. The selected processes represent two distinctly different methods of foam generation and experiment types, designated as follows:

1. Foams produced in a melt cycle from a pre-dispersed compact without material agitation (methods 1 and 6).
2. Foams produced by a dynamic process (liquid agitation), for which method 5 has been adopted as a model process.

The experiments with predispersed material (1) are discussed in this section (7.5), and those covering dynamic foaming in the subsequent section 7.6.

**7.5.1 Process Definition and Objectives.** The experiments defined in this section represent the preparation of plain and reinforced controlled density materials as metal foams from a compacted composite of the matrix metals and pre-dispersed solid foaming agents or pre-dispersed gas in the form of micro-balloons. For the following experiment definition, the compact foaming with a predispersed foaming agent has been selected as the representative process, since it involves the gas generation as part of the low-g processing. The use of microballoons is merely considered as a back-up experiment. The equipment and processing requirements for both methods are identical; the processing time requirements are more stringent for the selected experiment method, so that it automatically covers the back-up method.

The objectives of this experiment group are:

1. The investigation of the criteria for, and the control of, stable gas dispersion in liquid metals which can only be obtained under low-g conditions.

2. The effect of reinforcements upon dispersion stability.
3. The definition of criteria for foam (gas bubble) size control.
4. To evaluate the effectiveness of various foaming agents.
5. The preparation of a controlled density material which can be evaluated for properties and applications.
6. To obtain data and experience on the management of expanding liquid-gas mixtures.

All necessary data and qualitative information are derived from the evaluation of the product material after low-g processing and correlation with measured low-g processing conditions. (g -levels, processing temperatures).

7.5.2 Verification Requirements. The requirements for the verification of the characteristics indicated by the objectives, above, are:

1. Preparation of compacted pre-material samples with variation of
  - a. Foaming compounds, amounts and degree of dispersion.
  - b. For reinforced CDM, fiber material, configuration and content.
2. Specification of exact processing characteristics, particularly the low-g process phases and the temperature profile for optimum gas evolution.
3. Facilities for processing of the samples (1) under the conditions (2).
4. In-process measurement of material temperature and actual g-levels.
5. Product recovery and evaluation for the characteristics defined in 7.5.1 by
  - a. Measurement of bulk density.
  - b. Microstructural evaluation of gas and reinforcement distribution.
  - c. Metallurgical (metallographic) evaluation.
  - d. Chemical analysis of the effect of the generated gases upon bubble wall stabilization.
  - e. Measurement of mechanical properties, such as stiffness and strength.
6. Correlation of measurements (5) with processing conditions (4).
7. Assessment of practical applications of the obtained product properties.

7.5.3 Experimental Materials. For the purpose of the objectives defined in 7.5.1, there is a wide choice of base materials. The objectives could even be met with low-melting alloys (70 - 150° C) for which effective foaming agents have been defined (urea, oxalates). However, the use of base materials with higher melting point is more desirable, since they are representative of practical materials and generate more useful processing data. Two base materials have been selected:

1. Aluminum alloys.
2. Tin or Cu-Sn alloys.

The difference between these two material types is that in (1) gas generation starts in the solid state or the solid-liquid transition, whereas in (2) all gas is generated in the liquid state by overheating of the melt. The experimental evaluation is based on aluminum alloys since they represent the more stringent processing requirements. The use of aluminum further permits the use of the same preparation techniques, sample container materials etc. as defined for composites (7.3, 7.4). It automatically covers the processing requirements for Tin (2), since the max. processing temperatures are identical (700° C).

On the basis of prior studies, the following foaming agents have been selected:

Titanium Hydride ( $TiH_2$ )

Zirconium Hydride ( $ZrH_2$ )

Detailed data on the gas evolution vs. temperature and time are available from contract NAS8-27806. This study further identified the most promising fiber materials for reinforced CDM. The choice of either graphite, aluminum or silicon carbide fibers can be left open, since it does not affect the experiment requirements.

7.5.4 Material Quantity and Sample Configuration. Two sample types and sample container designs may be used: Either a fixed-size cylindrical container, 50% filled with compacted material and the remainder with a slightly pressurized high-purity argon, or an expandable cylindrical container with bellow-walls, 100% filled with compacted material. The first type permits an increase of the bulk material volume by a factor of 2, whereas the increase for the bellows container is limited to a factor of 1.6. In either case, the amount of material

is approximately the same as for fiber composite experiments (7.3, 7.4) with the standard 1.2 diam x 7 cm cylindrical sample, so that the heating requirements are comparable. The sample dimensions and material quantities are as follows:

	<u>Fixed Sample</u>	<u>Expanding Sample</u>
Diameter (I. D. ) (cm)	1.6	1.6 (mean)
Original Length (cm)	7	4
Expanded Length (cm)	-	6.4
Original Volume (cm <sup>3</sup> )	14	8
Final Volume (cm <sup>3</sup> )	14	12.8
Metal Volume (cm <sup>3</sup> )	7	8
Material Weight (Al-base) (gr)	20	23
Original Density (gr/cm <sup>3</sup> )	2.8	2.8
Final Density (gr/cm <sup>3</sup> )	1.4	1.75
Material Heat Content (30-700° C) (cal)	4,800	5,500
Heat Content incl. Container (cal)	7,400	8,100

7.5.5 Experimental Process Definition. The preparation, performance and evaluation of low-g experiments comprises the following operational phases:

1. Preparation of the component materials, dosaging for exact composition, dry mixing and compaction into the sample container. During all these operations, carried out in the laboratory, the materials have to be kept under uninterrupted protection against oxidation by a high-purity argon atmosphere. Preparation of the materials includes the preparation of the matrix powder and surface treatments of both matrix and reinforcements.
2. Installation of the (sealed) sample capsule in the experiment apparatus and the low-g test facility.
3. Low-g processing, consisting of heating through matrix-melting to the processing temperature of 700° C. hold at this temperature for a pre-determined period, followed by induced cooling through solidification. Measurements during this melt cycle are sample temperature and g-loads, both vs processing time.

4. Sample recovery and removal from container.
5. Sample evaluation, consisting of photomicrographic analysis, strength tests and correlation of results with the sample composition (1) and the in-process measurements (3).

The sequence of the individual operational steps is identified in the Process Flow Diagram, Fig. 7.5-1. (Bold frames indicate g-sensitive process phases; dotted frames indicate optional phases).

#### 7.5.6 Low-g Test Requirements

The g-sensitive processing time extends from the time at which the material adjacent to the sample wall approaches the melting temperature (app. 600° C for Al-base and 200° C for Sn-base), to the time of completed material solidification. For Sn-base material, the heating rate is immaterial, since gas generation starts above the melting temperature. For Al-base, the heating rate has to be as high as possible to minimize gas evolution while the matrix is still in the solid state. Time requirements are again based on aluminum samples since the requirements are more stringent and automatically cover lower melting alloys.

7.5.6.1 Heating Method. Since the gas evolution, starting at low rate at app. 250° C increases with temperature and time (see report GDCA DBG73-001, pp. 3-93 to 3-101) accurate control of heating rate is required which can only be obtained with electrical furnaces. Furthermore, rapid cooling is required to prevent collapse of the gas filled voids, at least for sample type (a), (7.5.4). This can best be achieved with the electrical furnace defined in 6.2.1.2 and Fig. 6-4 which includes the closed cooling system (6.3.2.2) and Fig. 6-17.

7.5.6.2 Time-Temperature Program. The optimum times for heating and cooling have been calculated as follows:

Preheating I to 210° C (ground)

Heating rate	Optional
Hold time at 200° C	300 sec
Hold power at 200° C	200 watts



Heating II to 510° C

Heating rate	10° C/sec
Power input	1,600 watts
Heating time II	30 sec

Heating III - Melting (510 - 660° C)

Mean heating rate	4° C/sec
Power input	1,200 watts
Melting time III	60 sec

Heating IV - to 700° C

Heating rate	4° C/sec
Power input	600 watts
Heating time IV	15 sec

Heating V - hold at 700° C

Power input	400 watts
Hold time V	15 sec

Cooling VI through solidification

Cooling rate (mean)	10° c/sec
Water flow rate	40 cc/sec
Time to solidify	20 sec

Cooling VII below 600° C

Cooling rate	undefined
Water flow rate	30 cc/sec
Cooling time VII	(to payload landing)

7.5.6.3 Total Low-g Time. The total g-sensitive processing period consists of Phases III to VI, above and amounts to:

Total Low-g Time	100 sec.
------------------	----------

In addition, 60 sec of Phase I and all of Phase II may be placed in pre-zero-g test (flight) period (90 sec for RR1-Trajectory A).

7.5.6.4 g-level. During the total low-g period defined above, any g-forces acting on the sample should be  $10^{-5}$  g (desirable) or at least  $10^{-4}$  g (acceptable).

7.5.7 Low-g Facilities and Experiments. The total low-g time of 100 sec places the experiments in the typical rocket regime. As shown below, a maximum of 3 experiments can be carried out on one flight of RR1 - Trajectory A (standard WSMR).

7.5.7.1 Payload Limitations. Using the equipment defined in 7.3.8, the available payload weight and space are ample for 3 experiments. The limitation to 3 experiments is solely determined by power requirements. Only with the following sequencing of experiments and process phases (I - VI) can the power consumption be kept within the max. output capability of the support module of 2,800 watts (numbers in chart identify experiments 1, 2, 3):

<u>Process Phases</u>	I	II	III	IV/V	VI	<u>Total</u>
(sec)	X	30	60	30	20	<u>Power</u>
(watts)	200	1600	1200	600	0	<u>Watts</u>
<u>Flight time (sec)</u>						
0-60	1, 2, 3					600
60-90	2, 3	1				2,000
90-150	2, 3		1			1,600
150-180	3	2		1		2,400
180-210		3	2		1	<u>2,800 (max)</u>
210-240			2, 3			2,400
240-270			3	2		1,900
270-300				3	2	600
300-320					3	-0-

According to this schedule, the total low-g processing time extends from 90 to 310 flight seconds (limit 333 sec.) and amounts to 220 sec (max 243 sec.).

7.5.7.2 Experiment Definition. It is concluded on the basis of the foregoing evaluations that predispersed CDM experiments can be effectively carried out on research rocket class 1, Trajectory A. The use of electrical furnaces is mandatory. A total of 3 experiments can be accommodated per flight. The total required low-g time is well within the available low-g time of RR-1, as evidenced by the following experiment definition:

Low-g Facility	RR 1
Trajectory	A (Standard WSMR)
Available Low-g Time	243 Sec.
Number of Experiments	3
Required Total Low-g Time	220 Sec.

7.5.8 Apparatus and Payload Definition. The payload consists of the support module and three (3) processing modules representing the apparatus. No other equipment, such as gas supply for atmosphere control is required.

7.5.8.1 Processing Modules. The processing module consists of an electrical furnace with resistance heating element operating at 1250° C, and a closed (circulating) water cooling system. The heating system is described in Sect. 6.2.1.2 and the cooling system in Sect. 6.3.2.2. The module assembly is illustrated in Fig. 6-4. Its major physical and functional data are:

Dimensions	24 diam. x 16 cm high
Weight (with coolant)	13.5 kg
Max. Power Rating	1,800 watts
Max. Power Required	1,600 watts
Power Consumption/Expt.	47 wh

7.5.8.2 Support Module. The required outfitting of the support module and the resulting weight and space requirements are as follows (numbers in parenthesis identify components specified in Sect. 5.2.2 and Table 5-1):

### Weights

Basic Structure (1, 2, 3)	33 kg
2 Batteries (4)	6 kg
Power Conditioning (5-A, B, C2)	12 kg
Sequencer, Recorder (6, 7)	4 kg
Contingency	2 kg
Total Weight	57 kg

### Experiment Space (Axial Height)

Net Space Available	80 cm
Required for 3 Expt. Modules	48 cm
Surplus	32 cm

### Power

Total Available	220 wh
Required for 3 Expts.	165 wh
Reserve/Surplus	55 wh

7.5.8.3 Payload Assembly. The payload assembly is illustrated in Fig. 7.5-2. The apparatus consists of 3 processing modules. Payload weight, dimensions and functional data are as follows:

### Payload Weight

3 Processing Modules (7.5.8.1)	40.5 kg
Support Module (7.5.8.2)	57 kg
Total Weight	97.5
RR-1 Payload Capability	130.0 kg
Surplus	32.5 kg

### Payload Dimensions

Max Diameter	38 cm
Max Height	150 cm
Payload Diameter	32 cm
Payload Height	120 cm

### Payload Dimensions (Cont'd)

A <sub>x</sub> ial Height for Apparatus	80 cm
Axial Height Required	48 cm
Surplus Space	32 cm

### Payload Power

Total Energy Available	220 wh
Required for Experiments	165 wh
Reserve/Surplus	55 wh

The surplus capabilities could be utilized for an additional experiment provided that it either requires no significant power, or that its power requirements are scheduled before or after 150-240 flight seconds, during which period the power rate of the CDM experiments reaches the max. capacity of the support system (2,800 watts).

The use of the power surplus for pre-flight preheating from the batteries rather than from the ground is unfeasible since the total power required for pre-heating and soaking of the expt. chamber of 117 wh far exceeds the surplus.

### 7.5.9 Experiment Performance

7.5.9.1 Ground Operations. Operations at the launch site prior to count-down consist of:

1. Dry payload check-out
2. Filling of coolant tanks (water)
3. Second check-out (functional, measuring)
4. Vehicle installation
5. Final checkout through ground support plus

operations during count-down:

6. Preheating with ground power for a min. of 600 seconds.

7.5.9.2 Flight Operations. The sequencing of flight operations is pre-set (program tape) and is defined in the time diagram, Fig. 7.5-3.

7.5.9.3 Post-Flight Operations at the launch site consist of:

1. Payload recovery
2. Payload removal from vehicle section
3. Recovery of sample, recorder tape and telemetry records
4. Cleaning of payload assembly.

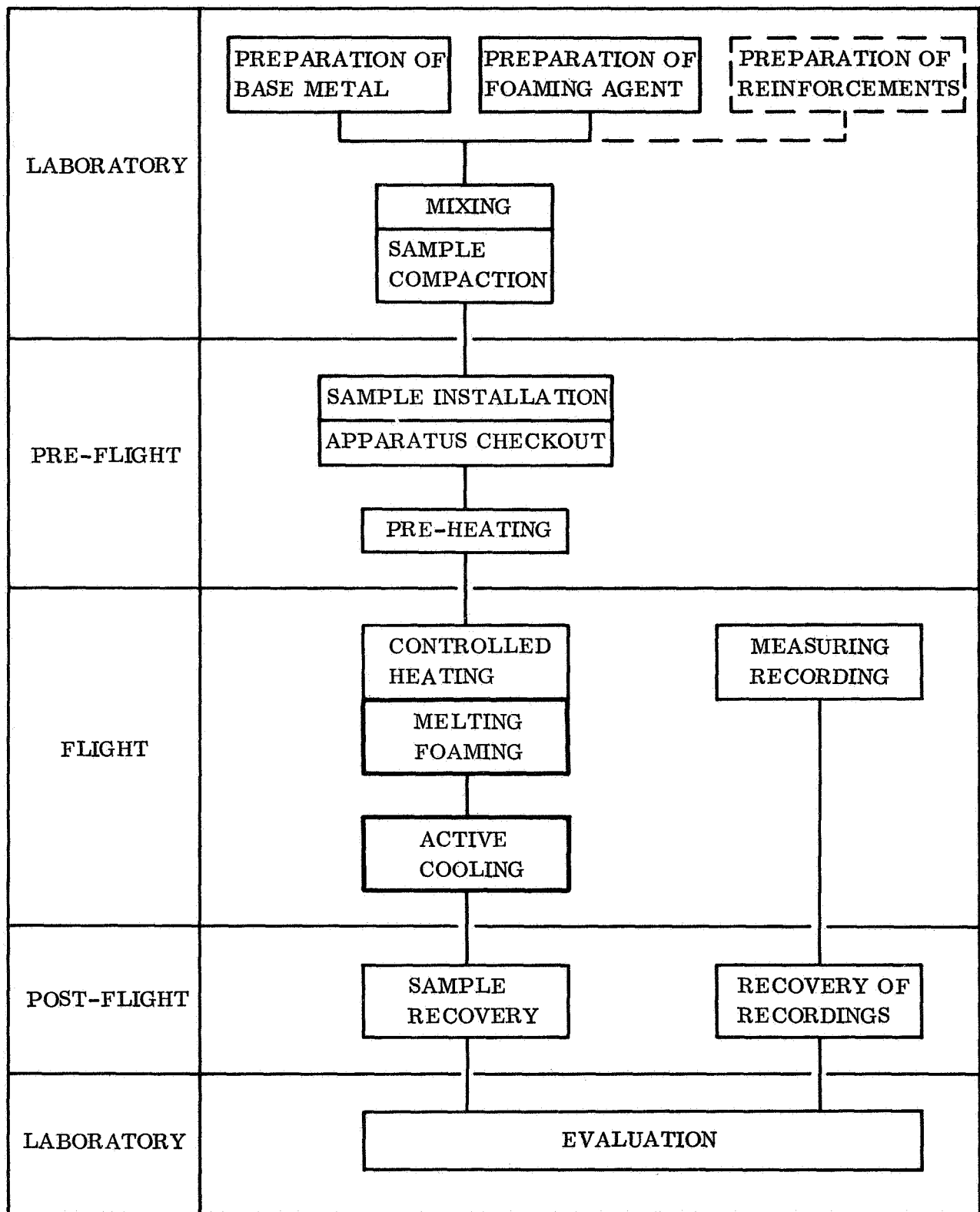


Figure 7.5-1. Process Flow Diagram - Controlled Density Metals - Predispersed Compact (Thermal Foaming)

## ROCKET PAYLOAD ASSEMBLY

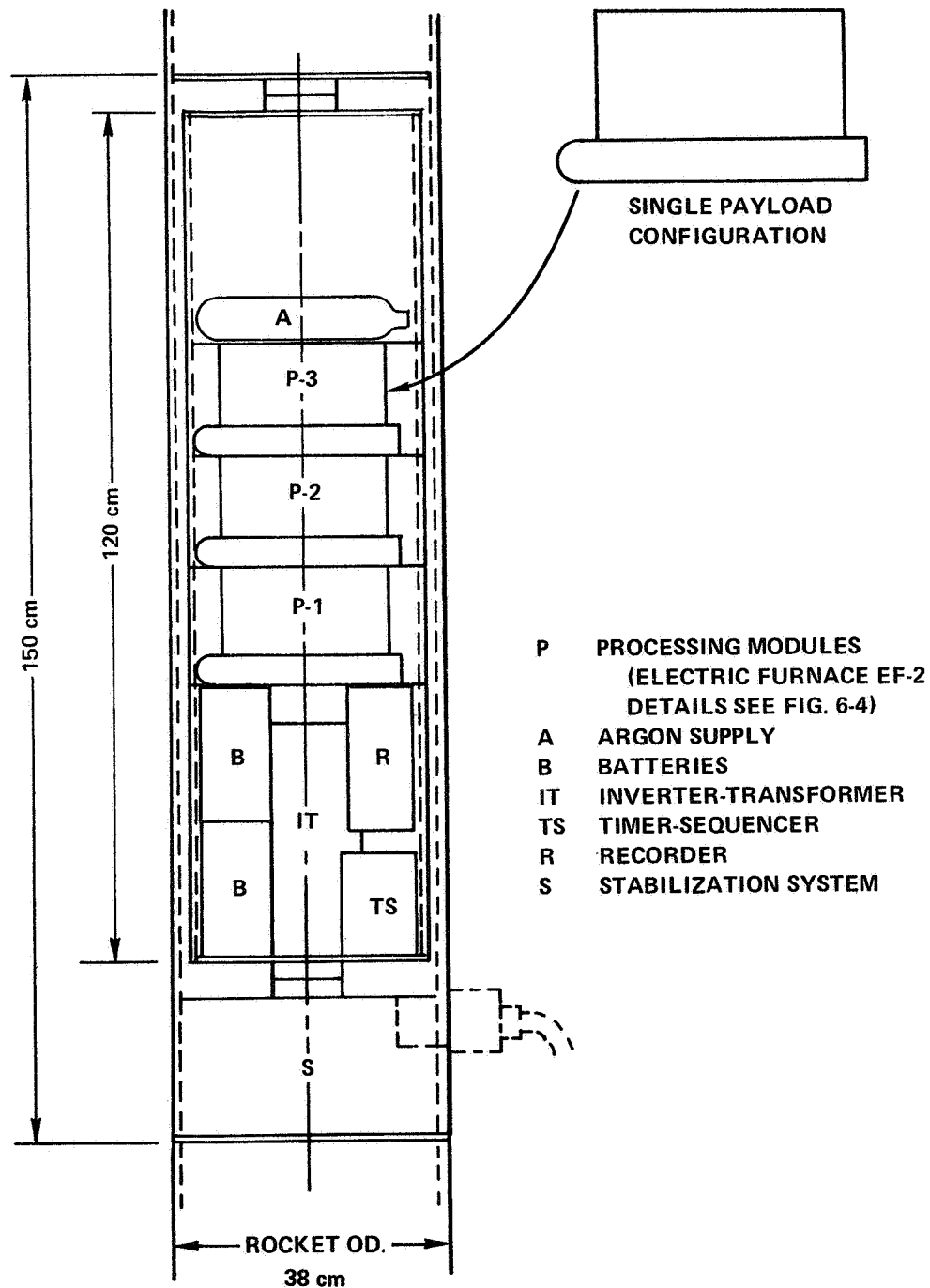


Figure 7.5-2. Payload Assembly — Controlled Density Metals — Predispersed (Thermal Foaming)



# EXPERIMENT PROGRAM – TRAJECTORY A

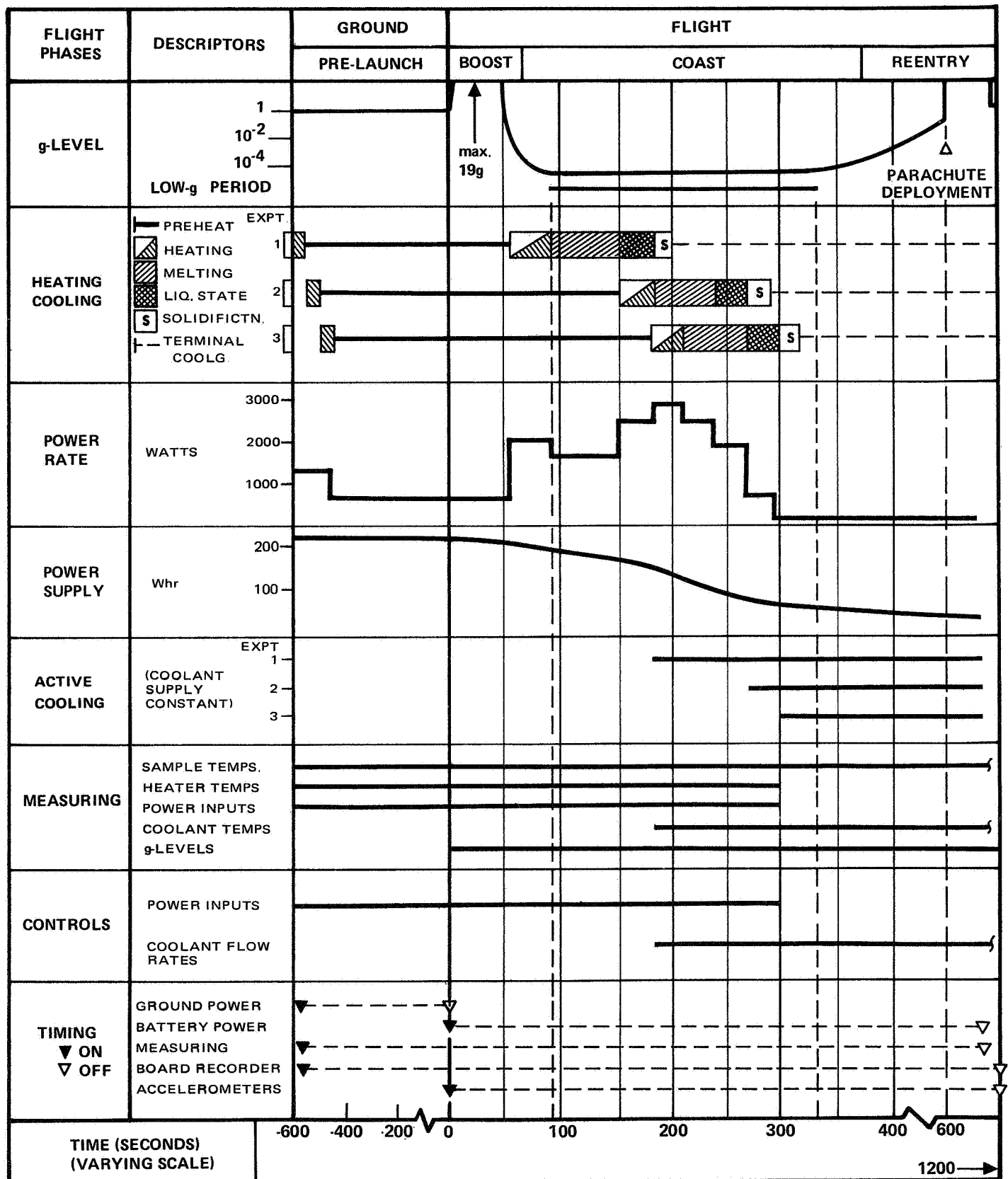


Figure 7.5-3. Time Diagram - Controlled Density Metals - Predispersed

## 7.6 CONTROLLED DENSITY METALS - DYNAMIC FOAMING

A general discussion of controlled density metals (CDM) and the methods of foam generation is presented in the introductory part of Section 7.5.

### 7.6.1 Process Definition and Objectives

Methods of dynamic foam generation in molten metals are: (1) gas injection foaming (2) cavitation foaming and (3) ultrasonic foaming. As discussed in the introductory part of Section 7.5, and for the reasons stated there, cavitation foaming (2) has been selected for initial experiments. In this method, gas is dispersed in the molten metal by violent mechanical mixing of a premeasured amount of liquid metal and gas in a container. Reinforcement may be added to the metal to obtain a reinforced foam. The objectives of low-g experiments are:

- (1) To verify the feasibility and effectiveness of the process, possible only under low-g conditions.
- (2) To determine the criteria for foam (gas bubble) stability and for deceleration to zero - motion after agitation.
- (3) To investigate the effect of reinforcements upon foam stability and product properties.
- (4) To obtain a product which can be evaluated in the laboratory
- (5) To generate data and experience with regard to process techniques and equipment design.

All necessary data and qualitative information are derived from the evaluation of the product material after low-g processing and correlation with measured low-g processing conditions (g-levels, processing temperatures). Observation and recording of the dynamic behavior of the material during agitation would be highly desirable; it has, however, been excluded in view of the involved equipment complexity. It is expected that the material behavior during liquid-state processing can be reconstructed from the characteristics of the solidified product.

### 7.6.2 Verification Requirements

The requirements for the verification of the data and characteristics implied by the stated objectives are:

- (1) (Materials Parameters): Preparation of samples with varied combination of:
  - (a) Base alloy composition
  - (b) Gas composition
  - (c) Metal/gas ratio
  - (d) Reinforcement type and content
- (2) (Process Parameters):
  - (a) Specification of exact processing conditions and procedures
  - (b) Variation of agitation mode (shape and velocity of stirring elements)
- (3) (Apparatus): Facility for the processing of the samples (1) under the conditions (2).
- (4) (Measurements): In-process measurement of material temperature and actual g-levels.
- (5) (Evaluation): Sample recovery and evaluation of:
  - (a) Foam dispersion and cell size
  - (b) Reinforcement dispersion and interaction with (a)
  - (c) Effect of gas composition on gas cell stability (surface stabilization by chemical reaction)
  - (d) Metallurgical characteristics, as related to (a), (b) and temperature profile (heating and cooling).
  - (e) Obtained material bulk density
  - (f) Mechanical properties of the bulk material
  - (g) Stiffness and strength to density ratio, from (e) and (f).
  - (h) Correlation of all evaluation data with materials and processing parameters, (1) and (2), above.
  - (i) Reconstruction of foaming effectiveness of the agitation device and the mixture behavior during processing.

### 7.6.3 Experimental Materials

7.6.3.1 Matrix Materials. While lower melting alloys, such as Sn-Cu alloys may also be considered for these experiments, preference is given to aluminum for the following reasons:

- (1) Practical usefulness
- (2) Moderately high processing temperature
- (3) Existing data from prior work, particularly with regard to optimum gas composition
- (4) Same processing temperature and, consequently, adaptability to same equipment as 7.3 - Fiber/Particle Composites.

Specific alloys may be used, such as Al-Cu-Si alloys. This does, however, not affect this evaluation. For applicable data, see Section 7.3.3 (1).

7.6.3.2 Gases. According to prior laboratory experiments, the most effective gas for foam generation is the following gas composition:

Argon 98% - Oxygen 2%

This composition provides just sufficient oxygen to fortify the gas cell walls by surface oxidation.

7.6.3.3 Reinforcement Materials. For reinforced CDM experiments, only short fibers with a length of app. 1 mm will be used (compatibility with liquid mixture agitation). Applicable fiber materials are listed in Section 7.3.3(2). Experiments carried out with  $Al_2O_3$  whiskers showed that they do not break, even at violent high-speed mechanical agitation, as long as the matrix exhibits a low viscosity (liquid metal viscosity regime).

### 7.6.4 Material Quantities and Sample Configuration

The material quantity is related to the sample configuration which, in turn, is dictated by flow pattern of the liquid-gas mixture during agitation.

A study of the mixture flow pattern obtained with various types of agitating devices and agitation modes indicated that a cylindrical sample with a length to diameter ratio between 2 and 3 represents an optimum sample shape with regard to gas dispersion and adaptability to a variety of foam generation techniques. The ultimate choice of technique and the related configuration of the agitating element (stirring or oscillating element) is left open, since it requires fluid mechanics studies and laboratory experiments which go far beyond the scope of this study. As a model, sufficiently representative of all potential techniques, a single rotating (or alternately rotating) agitator, driven with 28V-DC motor has been selected for this evaluation.

Considering limitations as to heating and power requirements, a cylindrical sample of 2.5 cm diameter x 6 cm long ( $L/D = 2.4$ ) has been selected. The sample consists of the cylindrical container, the solid material and the gas. The metal is cast into the container along the cylindrical wall in form of a hollow cylinder whose thickness is designated as  $t$ . For various gas contents (in % of the total sample volume) the material quantities, weights and the value  $t$  are as follows:

<u>% gas</u>	<u>Metal Vol. (cm<sup>3</sup>)</u>	<u>Metal Weight (gr)</u>	<u>t(cm)</u>
30	20.6	57	0.58
40	17.6	46	0.46
50	14.7	41	0.37
60	11.8	33	0.28

The original metal arrangement may vary for other agitation techniques. This does, however, not affect the essential experiment requirements (heating, low-g time, equipment).

#### 7.6.5 Experiment Process Definition

Processing comprises the following major phases:

- (1) Preparation of the component materials, mixing and casting into the sample container in a high purity argon atmosphere.

- (2) Attachment of the agitating element and sealing of the sample in an atmosphere of the foaming gas as defined in 7.6.3.2.
- (3) Installation of the sample and the apparatus.
- (4) Sample processing consisting of the following phases:
  - I. Pre-heating to 500° C
  - II. Melting (500-700° C)
  - III. Foaming for 10 seconds
  - IV. Deceleration of the agitated mixture (30 sec)
  - V. Solidification
  - VI. Terminal cooling

Phases II - V require low-g conditions.
- (5) Sample recovery and removal from container
- (6) Sample evaluation.

The sequence of the individual operational steps is identified in the Process Flow Diagram, Fig. 7.6-1. (Bold frames indicate g-sensitive process phases).

#### 7.6.6 Low-g Test Requirements

Foam generation requires accurate time-temperature control which can only be achieved with electrical heating.

7.6.6.1 Time and Power Requirements. For the processing phases defined in 7.6.5(4) the time and pertinent power requirements for the individual experiment are as follows:

		<u>Power/Watts</u>	<u>Time/Seconds</u>
I	Preheating to 500° C	1200	60
	Hold at 500° C	400	300 min.
II	Melting (500 - 700° C)	800	90*
III	Foaming (700° C)	600	10*
IV	Stillling (deceleration) (700° C)	500	30*
V	Solidification & 700 - 450° C)	100	120 min.
VI	Terminal Cooling	100	120 min.
	Total Flight Energy	49 Wh	
	Total Low-g Time (*)	7.6-5	150 sec

From the viewpoint of the basic process, phase II is not g-sensitive; it is, however, placed into low-g period in order to keep the melting material along the container wall in position, thus maintaining conductive heat transfer as the melting progresses radially toward the sample center.

7.6.6.2 g-Level. During the low-g period defined above, any g-forces acting upon the sample (other than those induced by agitation) should not exceed  $10^{-5}$  g (desirable) or  $10^{-4}$  g (acceptable).

#### 7.6.7 Low-g Facilities and Experiments

The required low-g time of 150 seconds places the experiments in the typical rocket regime. The number of experiments which can be accommodated in one flight is determined by the following evaluation of payload weight, space and low-g time limitations. The evaluation is based on rocket class 1, Trajectory A (Standard WSMR capability).

##### 7.6.7.1 Payload Weight Limitations (Equipment Data see 7.6.8).

Rocket Payload Capability	130 kg
Support Module	57 kg
Experiment Module (each)	14.5 kg
Total Payload 4 Expts.	115 kg
Total Payload 5 Expts.	129.5 kg

##### 7.6.7.2 Payload Space Limitations (Measured in axial height in payload can)

Available Space	80 cm
4 Expt. Modules	80 cm
5 Expt. Modules	(unfeasible)

7.6.7.3 Time and Power Limitations. Five experiments exceed the low-g time of rocket class 1-A (243 sec) as well as the max discharge rate of the power system

(3100 watts total, 2800 watts transformed). However, 4 experiments, also indicated by space considerations, can be accommodated by the following scheduling (numbers in chart identify processing modules 1 to 4):

<u>Low-g Time</u> <u>(Sec)</u>	<u>Hold/550° C</u> <u>(400 W)</u>	<u>Melting</u> <u>(800 W)</u>	<u>Foaming</u> <u>(600 W)</u>	<u>Solidific.</u> <u>(100 W)</u>	<u>Term. Coolg.</u> <u>(100 W)</u>	<u>Total Power</u> <u>(Watts)</u>
0-90	3, 4	1, 2				2400
90-130		3, 4	1, 2			2800
130-180		3, 4		1, 2		1800
180-220			3, 4		1, 2	1400
220-240				3, 4	1, 2	400
240-900					1, 2, 3, 4	400

7.6.7.4 Experiment Definition. It is concluded, that CDM experiments with dynamic foaming can be carried out effectively on rocket class 1 Trajectory A with electrical furnaces. A total of four (4) experiments can be accommodated per flight, operating in pairs, as evidenced by the following data:

Available Low-g Time	243 sec
Required Low-g Time	240 sec
Available Energy	220 wh
Energy Required for Experiments	194 wh
Total Required, incl. Support	219 wh

#### 7.6.8 Apparatus and Payload Definition

The payload consists of the support module and 4 processing modules representing the apparatus. No other equipment such as atmosphere control or pressurization systems, are required.

7.6.8.1 Processing Modules. The four processing modules are identical and consist of (1) the unit shown in Fig. 6-4 and described in Sections 6.2.1.2 (electrical furnace) and 6.3.2.2 (closed cooling system), and (2) the agitation system (DC motor) measuring 6 x 6 x 4 cm and attached at the upper chamber cover. The agitation system increases the unit height by 4 cm and its weight by 1 kg. The major data of the processing module are:



Diameter	24 cm
Axial Height	20 cm
Weight incl. Coolant	14.5 kg
Processing Chamber	5 diam x 12 cm
Max Furnace Temperature	1250° C
Cooling System	Closed (circulating)
Coolant Supply	2 liters

7.6.8.2 Support Module. The outfitting and weight of the support module are (numbers in parentheses identify components specified in Section 5.2.2 and Table 5-1):

Basic Structure (1, 2, 3)	33 kg
2 Batteries (4)	6
Power Conditioning (5-A, B, C-2)	12 kg
Sequencer, Recorder (6, 7)	4 kg
Contingency (8)	<u>2 kg</u>
Total Weight	57 kg

7.6.8.3 Payload Assembly. The payload assembly is illustrated in Fig. 7.6-2. The apparatus consists of four processing modules stacked so that the sample and rocket axes coincide. Major payload data are:

Payload Weight

4 Processing Modules	58 kg
Support Module	57 kg
Total Payload	115 kg
Rocket 1-A Capacity	130 kg
Reserve	15 kg

### Payload Dimensions

Max. Diameter	38 cm
Max. Height	150 cm
Payload Can Diameter	32 cm
Payload Can Height	120 cm
Apparatus Diameter	24 cm
Apparatus Height	80 cm

### Payload Power

Total Stored Energy	220 wh
Total Energy Consumption	219 wh

## 7.6.9 Experiment Performance

### 7.6.9.1 Ground Operations (same as 7.5.9.1)

7.6.9.2 Flight Operations. The sequencing of flight operations is pre-set (program tape) and is defined in the Time Diagram, Fig. 7.6-3.

### 7.6.9.3 Post-Flight Operations (same as 7.5.9.3)

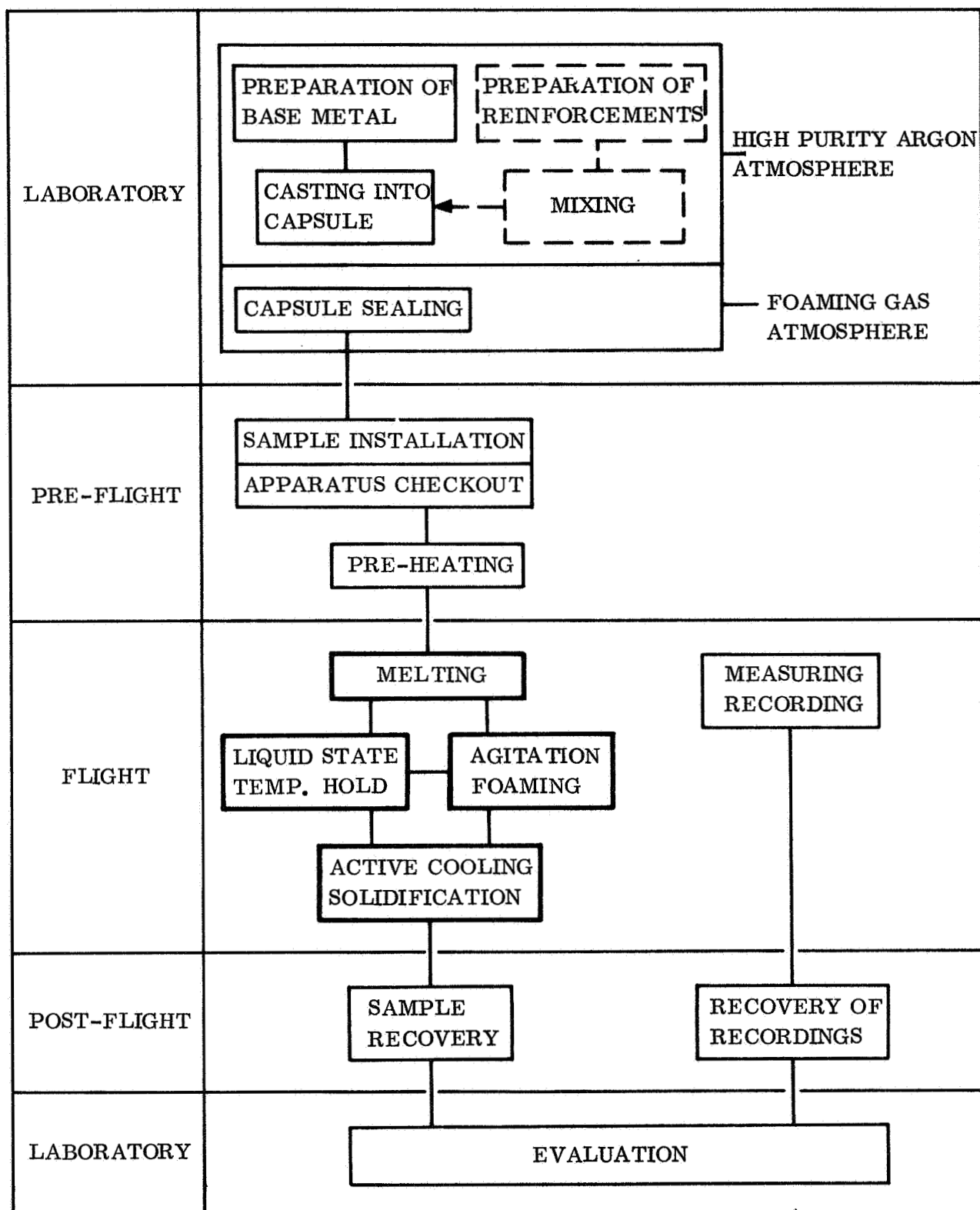


Figure 7.6-1. Process Flow Diagram - Controlled Density Metals - Dynamic Foaming

# ROCKET PAYLOAD ASSEMBLY

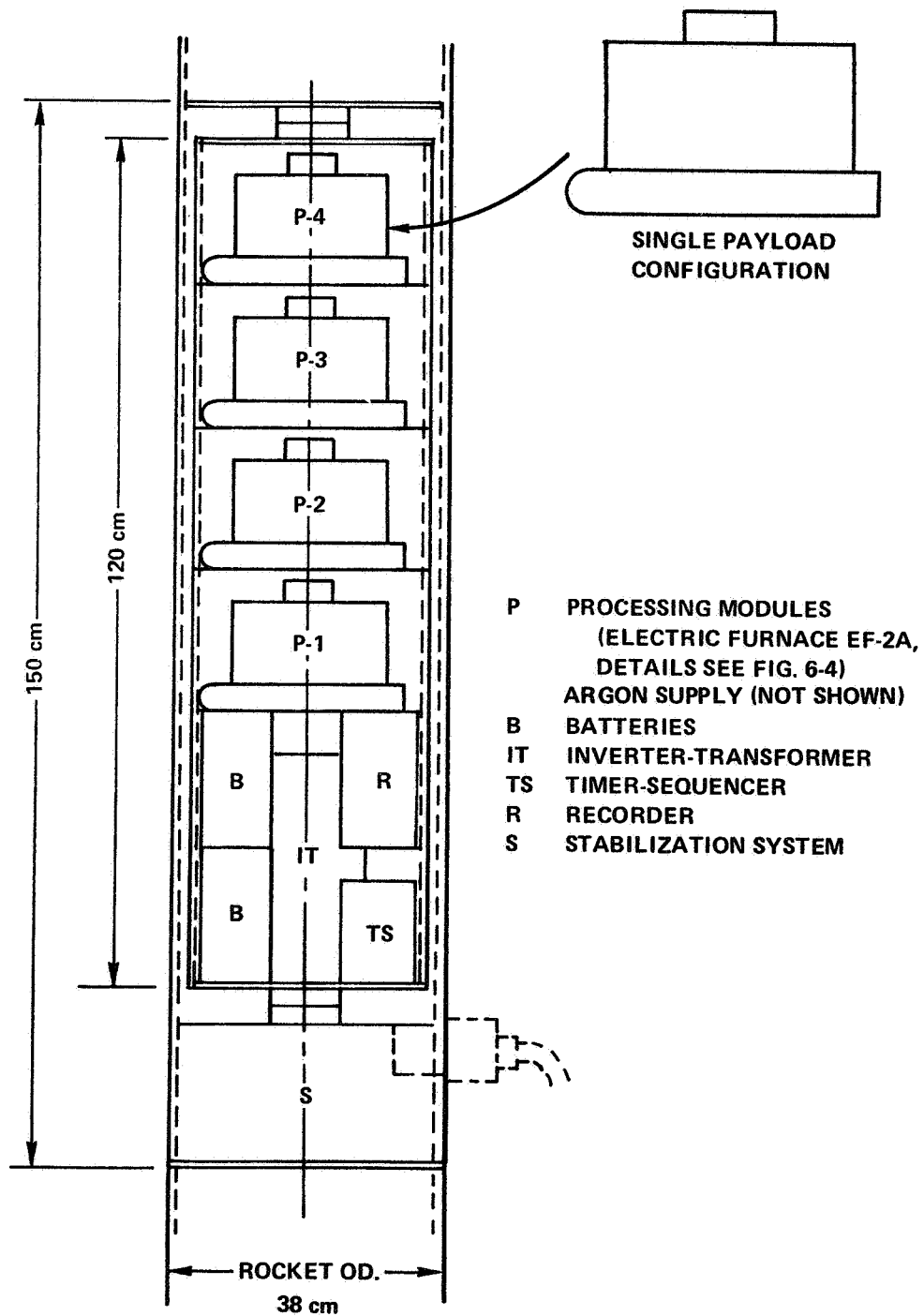


Figure 7.6-2. Payload Assembly — Controlled Density Metals — Dynamic Foaming

# EXPERIMENT PROGRAM – TRAJECTORY A

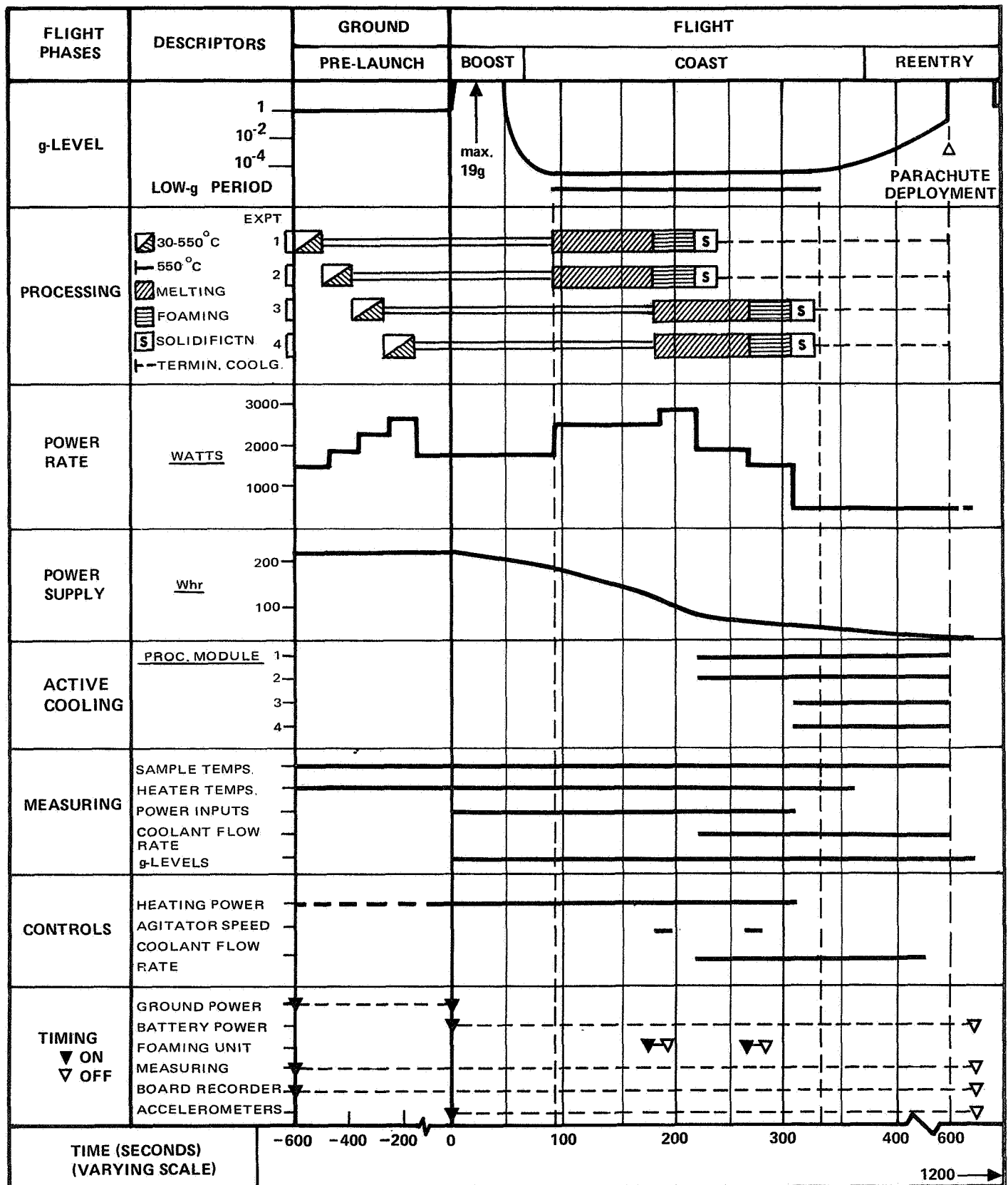


Figure 7.6-3. Time Diagram - Controlled Density Metals - Dynamic Foaming

## 7.7 UNIDIRECTIONAL EUTECTICS

### 7.7.1 Process Definition and Objectives

Under conventional isotropic cooling and solidification of eutectic alloys, the intermetallic phase precipitates in randomly distributed globules or lamellae. If, instead, cooling is induced from one end of the melt, a discrete solidification front moves slowly away from the cooled end; in this progressive solidification the intermetallic compound may precipitate in the form of continuous, unidirectional rods or lamellae. The production of such unidirectional eutectics is highly attractive for products which require high anisotropic strength, such as gas turbine blades.

The properties attainable in one-g processing (downward solidification) are very limited since the heat of fusion released at the solidification front produces violent convection currents that disrupt the continuity and directionality of the compounds. It is expected that under low-g conditions unidirectional properties approaching the theoretical value can be obtained, provided that three conditions are met: (1) high alloy purity, (2) unidirectional solidification and (3) low cooling or progression rate. The end product may be classified as a composite, consisting of the base metal matrix and unidirectional filamentary intermetallics.

Prime product characteristics are anisotropic mechanical, electrical and thermal properties. The strength in the direction of the intermetallic may differ from the random-oriented eutectic, as produced in one-g, by a factor of 2 to 4, and the elastic modulus up to a factor of 2, depending on the specific alloy and the precision of process control.

### 7.7.2 Verification Requirements

The prime criterion for the preparation of such composites in low-g experiments is the required processing time which, in turn, is determined by the solidification (progression) rate and the size of the product. For commercial high-purity alloys, the required progression rate is in the order of 1 cm/hr. It can be well seen that at this rate the

preparation of a 1-cm section of unidirectionally solidified material, which is considered a minimum size for evaluation, will take one hour, and the preparation of a sample which can be subjected to tensile tests 6-8 hours. Such experiments call for orbital facilities, such as the shuttle-based MS/MS laboratory.

However, for specially prepared small alloy samples of ultra-high purity the progression rate can be increased to 10-20 cm/sec. At this rate, a processed material section of 1 cm length can be well obtained in rocket experiments. We may, therefore, establish two verification levels:

Verification Level I: Preparation of small samples for metallurgical evaluation only, using specially-prepared ultra-high purity alloys.

Verification Level II: Preparation of larger samples for strength evaluation or prototype products using ultra-high or commercially high purity materials.

Rocket experiments are limited to verification level I.

### 7.7.3 Experimental Materials.

A number of eutectic alloys have been evaluated experimentally or suggested in the literature for structural, optical, electronic and magnetic applications. The most promising candidate material systems, together with their melting temperatures and their theoretically predicted ultimate strengths are as follows:

<u>System</u>	<u>Melting Temp ° C</u>	<u>Theoretical Strength (psi)</u>
Al-Al <sub>3</sub> Ni	627	47,000
Al-CuAl <sub>2</sub>	548	75,000
Ta-Ta <sub>2</sub> C	2800	295,000
Nb-NbC	2600	475,000

It is proposed to confine initial low-g experiments to the Al-base systems for the following reasons:

- (1) The comparatively low processing temperatures permit an accurate control of the solidification rate.
- (2) The experiment result will provide a basis for the definition of the processing parameters and techniques for experiments with high-melting alloys.
- (3) They can be carried out in the same apparatus used for experiments 7.12 and 7.13.

#### 7.7.4 to 7.7.9

Sample configuration, experiment requirements, low-g facilities, apparatus and experiment performance procedures are identical to 7.12 (Single Crystal Growth) and as described in Sections 7.12.4 to 7.12.9. The essential data are summarized in the following experiment specification.



## 7.8 SUPERCONDUCTORS

### 7.8.1 Process Definition and Objectives

The state of art of superconductors is still far away from the practical goal: to raise the superconducting temperature into a regime where the cost for maintaining the cryogenic temperature becomes economically acceptable. The achievement of this goal would be of vast technological and economical significance, in power generation (nuclear fusion), power transmission and all types of electrical apparatus or machinery.

Inspired by this potential, extensive research has been carried out world wide, particularly during the past ten years. However, most of the 2000 so-far discovered superconducting material compositions exhibit transition temperatures below 10° K, and only a limited number in the range from 10-20° K. The highest known transition temperature ( $T_c$ ) is 21° K found in ternary alloy of niobium, aluminum and germanium, achieved only in the form of small samples of laboratory-prepared material.

A technological and economical payoff can be realized only if both of the following requirements are met: (1) a transition temperature in the order of 25° K or more and (2) an alloying process adaptable to reasonable quantity production. In spite of all research efforts, the development of superconductors is still short of this goal and has, for all practical purposes, arrived at an impasse.

Experimental research has shown that the so-called beta-tungsten ( $\beta$ -W) crystal structure is the most favorable for the occurrence of high-temperature superconductivity. This crystal structure has the form of a compound  $A_3B$ , consisting of linear chains of atoms of a transition metal A, and B atoms of a non-transition element. Although the exact mechanism responsible for enhanced transition temperature is not known, the following requirements have been established empirically:

- (1) High percentage of, or complete, beta-tungsten crystal structure
- (2) Electron per atom concentration in the range 4.50 to 4.75
- (3) Perfect stoichiometry
- (4) A high degree of order, with possibly even-ordered superstructures of non-transition elements

A perfect combination of these conditions has never been achieved, since all terrestrial preparation methods are subject to gross segregation, affecting both the stoichiometry and the degree of order, and to micro-segregation in the form of coring, reducing the percentage of useful beta-tungsten structure. A reduction of these negative effects has been achieved by elaborate preparation techniques; however, such techniques are unfit for practical material production.

Since in zero-g segregation effects would be absent, it is to be expected that a high perfection of the crystal structure and a substantial increase of the transition temperature can be achieved, even using simple melting techniques for alloying. The basic validity of this contention has been proven in short-time low-g experiments carried out recently on the KC-135 research aircraft with several vanadium-base alloys. Even though the experiment conditions were imperfect in many ways, a pronounced improvement over terrestrially prepared material was obtained in a single melt cycle. The performance of zero-g experiments under more perfect processing conditions is, therefore, indicated. The objectives of such experiments are:

- (1) Demonstration of the effectiveness zero-g processing upon transition temperature, using known superconducting alloys.
- (2) Definition of the materials parameters responsible for high-temperature superconductivity as a basis for the development of advanced compositions.
- (3) Definition of critical processing parameters and optimized processing conditions.

#### 7.8.2 Verification Requirements

The gain obtained by zero- or low-g processing is ultimately verified by the difference in transition temperature for identical material compositions processed under equivalent conditions in one-g and zero-g.

To achieve the specific objectives stated in 7.8.1 and to satisfy the postulation for a process adaptable to quantity production, the following specific verification requirements have to be met:

1. Processing Requirements:
  - 1a. Alloying by a single melt cycle
  - 1b. Pressurized inert gas environment to suppress vaporization of constituents.

## 2. Verification of material properties:

- 2a. Percentage of superconducting crystal structure
- 2b. Lattice parameter
- 2c. Stoichiometry
- 2d. Degree of order
- 2e. Presence of segregated phases
- 2f. Degree of coring
- 2g. Presence of voids or porosities
- 2h. Transition temperature.

All property measurements (2) are carried out on the processed (finished) material and no in-process measurements are involved.

Properties (2a) through (2g) are verified by x-ray diffraction studies and by microstructural (metallographic) evaluations, for which only small sample quantities are required; they permit a tentative prediction of the transition temperature. More desirable is, of course, a direct measurement of the transition temperature (2h); this, however, requires larger sample quantities.

Consequently, experiments can be designed for two verification levels:

### Verification level I:

Verification of improvements in microstructural characteristics responsible for superconductivity by x-ray diffraction and microstructural measurements; correlation with processing parameters and prediction of transition temperature.

### Verification level II:

Direct measurement of the transition temperature and definition of its dependency upon materials- and process parameters on the basis of x-ray diffraction and microstructural measurements.

## 7.8.3 Experiment Materials

The selection of materials for zero-g experiments has been based on the following criteria:

- (a) Terrestrially achieved transition temperature over 15°K
- (b) Promise of transition temperature improvement
- (c) Thoroughly explored alloy systems and availability of applicable data
- (d) Reasonable melting temperature.

Criterion (a) eliminates the vast majority of known superconductor materials whose presently attainable low transition temperature renders the probability to ever reach the target temperature of 25° K very unlikely. Two alloy systems which meet all criteria are niobium and vanadium base alloys. Niobium alloys are presently at the top of the list of superconductors with a maximum transition temperature of 21° K. Their melting temperature is in the order of 2200–2400° C which is high, yet still acceptable.

The presently attainable transition temperature of vanadium-base alloys is somewhat lower (17° K), a shortcoming which is offset by the experimental advantage of a lower melting temperature (1800–2000° C).

The following alloys of these base metals appear most promising and have, therefore, been selected as model systems for the definition of low-g experiments (composition in ratios of atomic weight):

<u>Alloy</u>	<u>Present T<sub>c</sub> max</u>
<u>Niobium-Base</u> (melting temp ~ 2200–2400° C)	
Nb <sub>3</sub> Al <sub>.75</sub> Ge <sub>.25</sub>	21 ° K
(Nb <sub>3</sub> Al) <sub>4</sub> Nb <sub>3</sub> Ge	20.5° K
Nb <sub>3</sub> Ga	20° K
Nb <sub>3</sub> Al	18.8° K
Nb <sub>3</sub> Sn	18.1° K
<u>Vanadium Base</u> (melting temp ~ 2000° C)	
V <sub>3</sub> Si	17° K
V <sub>3</sub> Ga <sub>.25</sub> Si <sub>.75</sub>	17° K
V <sub>3</sub> Ga <sub>.5</sub> Si <sub>.5</sub>	16° K

#### 7.8.4 Material Quantities, Sample Size and Heating Methods

These characteristics have to be assessed jointly since their optimization can only be achieved by mutual trade-off.

7.8.4.1 Target Material Quantity. The final material quantity after processing is determined by the minimum quantity required for the sample evaluation measurements. For the x-ray diffraction and microstructural measurements of verification level I, a near-equidimensional sample is desirable. Since the final sample configuration after melting will very likely be spherical, all data are defined for spheres. The desirable sphere diameter for verification level I is 0.3 cm, however a diameter of 0.25 cm is acceptable as an absolute minimum.

The minimum final sample size for verification level II is either a sphere with a minimum diameter of 0.4 cm for the inductance method, or a rectangular sample of 0.25 x 0.25 x 0.4 cm for direct conductivity measurements. The latter sample can be cut from a sphere of 0.45 cm diameter, leaving sufficient material for x-ray and microstructural measurements. The 0.45 cm diameter sphere has been adopted as base value, since it permits the evaluation by both the inductance and conductivity measuring method. The minimum final sample sizes and material quantities are therefore as follows:

Verification Level	I	II
Shape	Sphere	Sphere
Dimension (cm)	0.25 diam	0.45 diam
Volume (cm <sup>3</sup> )	0.0081	0.047
Weight-V-base (gr)	0.041	0.235
Weight-Nb-base (gr)	0.058	0.342

7.8.4.2 Heating Method. The criteria for the choice of the heating/melting method are: (1) contact-free liquid material suspension, except contact with the solid sample portion; (2) no outgassing of constituents, except at the surface; (3) generation of sample configurations postulated in 7.8.4.1.

Various heating methods have been evaluated qualitatively and quantitatively. Most desirable would be induction heating in a free suspension system; it is, however not considered at this time because of the extensive equipment and control requirements and the undefined availability of an operational coil system. Numerical assessments of electric discharge techniques indicated the following disadvantages: (1) Excessive equipment requirements; (2) Control limitations; (3) Potential excessive material vaporization (4) Necessity of a wire of high L/D ratio, resulting in multiple spheres or odd-shaped pieces of inadequate size upon melting.

Resistance heating by a high-frequency current was found most effective and was, therefore, adopted for the experiment evaluation. It satisfies all the requirements stated above, is comparatively uncomplicated and can be easily controlled. The sample is a part of the secondary single-turn winding of a specially-built transformer whose primary coil is fed from a solid state inverter, converting the standard 28 volt battery supply into a high-frequency AC.

7.8.4.3 Sample Configuration and Size. The basic sample configuration is shown in

Fig.7 8-1a. Upon melting either of the following may occur:

- (a) The sample stays intact, center section deformed by surface tension.
- (b) The center section separates and forms 2 semi-spherical samples (Fig. 7.8-1b).
- (c) The center section breaks apart into a free sphere and two end pieces as in (b) (Fig. 7.8-1c).

Condition (c) is most desirable. According to studies, this condition can be achieved with an L/D ratio of 4. To obtain the evaluation sample quantities defined in 7.8.4.1, the following original sample quantities and dimensions are required:

<u>Verification Level I</u>	<u>Original Sample</u>	<u>Resulting Evaluation Samples</u>	
Shape	Cylindrical	Sphere	Half-Sphere
Number	1	1 and	2
Diameter (cm)	0.2	0.25	0.3
Length (cm)	0.8	-	0.15
Volume (cm <sup>3</sup> )	0.025	0.008	0.007
Weight V-alloys (gr)	0.125	0.040	0.035
Weight Nb-alloys (gr)	0.180	0.057	0.05

Verification Level II - Sample II

Shape	Cylindrical	Sphere	Half-Sphere
Number	1	1 and	2
Diameter (cm)	0.4	0.45	0.55
Length (cm)	1.6	-	0.3
Volume (cm <sup>3</sup> )	0.201	0.047	0.045
Weight V-alloys (gr)	1.0	0.24	0.23
Weight Al-alloys (gr)	1.45	0.34	0.32

## 7.8.5 Processing Phases

7.8.5.1 Pre-test (ground) operations comprise the preparation of the sample consisting of the following steps:

- (1) Preparation of powdered alloying constituents
- (2) Dosaging of constituents for accurate composition
- (3) Dry mixing
- (4) Sample compaction.

All operations are carried out in high-purity argon. Powder compaction is the commonly used method for sample preparation.

7.8.5.2 Test (low-g) operations represent the thermal (alloying) cycle consisting of

- (1) Heating and melting
- (2) Solidification by radiation cooling.

The thermal cycle has to be carried out in argon to prevent oxidation and at a pressure of 1-1.5 atm to minimize vaporization of alloying constituents. If other than the selected compaction method is used for sample preparation, an additional liquid-state agitation phase may be required to enhance alloy formation. For the selected sample preparation method this is not necessary, since alloy homogeneity is assured by the fine dispersion of powdered alloying constituents and thermal agitation during the melting phase.

7.8.5.3 Post-test operations consist of the preparation (cutting) of evaluation specimen and the evaluation measurements identified in 8.2.

7.8.5.4 The processing sequence is identified in the flow diagram, Fig. 7.8-2.

## 7.8.6 Low-g Test Requirements

7.8.6.1 Low-g Time Requirements. The definition of the low-g processing time necessarily has to be an optimum compromise between (1) adequate time to achieve alloying and (2) acceptable power and equipment requirements.

For the exploratory experiments of verification level I, an alloying time (melting range) of 1 sec is sufficient. For the more precise experiments of verification level II, the time of melting should be at least in the order of 10 seconds.

The power and equipment requirements for sample heating and melting are determined by the total thermal profile consisting of:

- (1) Heat absorbed by the heat content of the sample, i. e. solid-state heating and heat fusion.
- (2) Heat losses by radiation.
- (3) Heat losses at the sample ends.

Extensive numerical trade-off studies were carried out to arrive at an optimum combination of time at the melting temperature, acceptable heat losses and acceptable power requirements.

For the short times of verification level I, the end-losses can almost be neglected, and the heat requirements are primarily composed of sample heat content and radiation losses. The resulting data are stated below.

For the extended time requirements of verification level II, most of the energy output is lost at the sample ends (app. 55-70% of the total input). Sample heating accounts for app. 10-15%, and radiation for 20-25% of the total heat.

An optimized processing profile for verification level II, which serves as model for the low-g time definition, is shown in Fig.6-14. It is computed for the Nb-Al-base alloys with a melting temperature of 2200° C. It is also representative of the V-Si-base alloys whose somewhat lower melting temperature (2000° C) is offset by the higher heat of fusion.

The thermal profile (Fig. 6-14a) identifies a total heating time of 28 sec. The solidification time of the resulting free sphere is less than 2 seconds, placing the total processing time to 30 seconds. For some alloys, an optimum is obtained at a somewhat shorter or longer total time; a maximum total time of 40 seconds has therefore been adopted as experiment base value. To assure that none of low-melting constituents, such as gallium, is lost, all this processing time should be under low-g conditions.

The heat/power profile (Fig.6-14b) identifies a maximum power input of 700 watts. This can be easily achieved with the high frequency transformer identified in 8.4.2 and an output of 0.5 volts and 1400 amps. For each specific sample composition, the frequency, voltage and amperage have to be matched with the sample resistivity. The extremely low total energy requirements of 6 wh (=0.2 amp hrs for a 28-volt battery) do not warrant any preheating on the ground.



The maximum processing times and power requirements for verification level I and II are summarized below

Verification Sample	I	II
<u>Max. Time (seconds)</u>		
Heating/Melting	2.5	38
Solidification	0.5	2
Total Low-g	3	40
<u>Power/Heat</u>		
Max. Input, Watts	400	700
Total Energy (Wh)	0.25	6
Total Heat (Cal)	210	6000

7.8.6.2 Required g-Level. As outlined in 8.1, the prime purpose of low-g processing of superconductors is to eliminate or minimize gross- and micro-segregation of alloying constituents with a maximum density difference of  $6 \text{ gr/cm}^3$ . An accurate theoretical assessment of the relationship of segregation rate to g-level is not possible in view of the complex solid-liquid material system, its change with increasing temperature and the absence of applicable data. On the basis of the segregation data generated under contract NAS8-27806, experiences of one-g laboratory work and the experiences obtained in KC-135 experiments, the following max. g-levels have been defined.

Verification level I: A g-level of  $10^{-4}$  is desirable; however, a pronounced improvement of the alloy microstructure is expected at g-levels as high as  $10^{-2}$  g.

Verification level II: These experiments call for a high degree of perfection which can be obtained only at max. g-levels in the order of  $10^{-5}$  g.

#### 7.8.7 Low-g Facilities and Experiments

7.8.7.1 Correlation of Experiment Requirements and Facility Capabilities. A comparison of the required low-g processing times, above, with the low-g facility capabilities (Table 4-1) shows that type I experiments can be conveniently carried out in the MSFC drop tower, and type II experiments in a research rocket Class 1 or 2 and the minimum WSMR Trajectory A.

For drop tower experiments, a single experiment per drop is preferred; however the concurrent performance of two experiments is well within the payload capabilities of the drop package.

For verification level II rocket experiments, the sequential performance of several experiments is indicated by the relatively short processing time of 40 seconds, representing less than one-fifth of the net low-g flight time of 220 seconds.

The feasible number of consecutive experiments may also be limited by payload weight or space limitations. Using the apparatus data specified in 7.8.8, the values for four to six experiments per flight are checked against the payload limitations.

	<u>Low-g time</u>	<u>Weight</u>	<u>Height</u>
	(sec)	(kg)	(cm)
RR-2 Limitations	243	130	90
5 Experiments	200	115	70
6 Experiments	240	127	85

\*) Net axial height available for expts in payload can

The comparison shows that a maximum of 6 experiments can be carried out in sequence, leaving a time contingency of 3 seconds and a weight contingency of 3 Kg.

7.8.7.2 Definition of Facilities and Experiments. The two types of superconductor experiments, representing two verification levels, can be carried out effectively as follows:

#### Verification Level I

Low-g Facility	MSFC 300-foot drop tower
Number of Expts.	1 (or 2 concurrent) experiments per drop.
Required low-g time	3 seconds

#### Verification Level II

Low-g Facility	RR 1 or 2, trajectory A
Number of Expts	6 experiments in sequence
Required low-g time	240 seconds.

#### 7.8.8 Apparatus and Payload Definition

The experiment payload consists of one or several identical processing modules, an atmosphere control system and the support equipment.

7.8.8.1 Processing Modules. One complete processing module is used for each sample, so that each experiment can be individually controlled. The functional characteristics of the individual processing module are described in Section 6.2.3 and its design illustrated in Fig. 6-12. Identical modules are used for tower and rocket experiments in order to simplify control requirements and reduce cost. They differ only in the power rating and the size of the sample gripping heads. The major data are:

	<u>Tower Module</u>	<u>Rocket Module</u>
Max. Power Rating	500W	800W
Dimensions (cm)	16x18x14 high	
Weight	12 kg	12 kg

7.8.8.2 Rocket Support Module. The support module for rocket experiments is described in principle in Section 5. The specific outfitting and major data are as follows (numbers in parenthesis identify components specified in Section 5.2.2 and Table 5-1):

Basic Structure (1, 2, 3)	33 kg
1 Battery (4)	3 kg
Power Conditioning (5-A, 5-B)	6 kg
Sequencer, Recorder (6, 7)	4 kg
Contingency (8)	2 kg
	<hr/>
	48 kg
Net Space (in axial height) Available for Apparatus	90 cm
Total Power Capacity	110 wh.

7.8.8.3 Rocket Payload Assembly. The payload assembly is shown in Fig. 7.8-3. The six modules are stacked so that the samples fall into the rocket axis (min. g-level). Major payload data are as follows:

Weight and Space (by axial height)

	<u>Weight (kg)</u>	<u>Height (cm)</u>
Support Module	48	60
6 Processing Modules	72	84
Argon Supply System	<u>7</u>	<u>-</u>
Totals	127	144
R-1 Capacity	130	150
Contingency	3	6
<u>Power</u>	<u>Payload Capacity</u>	<u>Required</u>
Total Energy	110 wh	64
Max Discharge Rate	1500 w	900 w

In view of the substantial power surplus (46 wh), all check-out tests can be made with battery power and no ground support is required.

7.8.8.4 Drop Tower Assembly. In view of the modest weight and space requirements, an experiment assembly for one (or two concurrent) experiments can be easily installed in the drop tower package, defined in Section 4. and Fig. 4-1 . By the use of flight hardware, the tower experiments serve as check-out tests for payload components. In addition to the standard support equipment of the drop package, the following components are required:

1 Battery (4)	3 kg
Power Distribution (5A)	3 kg
Inverter with Controls (5B)	3.5 kg
Processing Module	12 kg
Argon System	<u>6 kg</u>
Total Weight	27.5 kg
Energy Consumption/Processing	0.3 wh
Energy Consumption/Support	3.5 wh
Total Consumption	4 wh

In view of the minimal power requirements, at least 8 experiments, including check-out tests, can be fully supplied by one battery charge (110 wh).

### 7.8.9 Experiment Performance

7.8.9.1 Pre-test (Ground) Operations. As discussed in Sec. 8.6.1, there is no need for ground-preheating and all power is supplied by the on-board battery. Ground operations consist then only in:

- (11) Pressurization of all processing chambers. Status-check.
- (12) Short power pulse - each expt. module to assure functioning of heating systems. Status-check.
- (13) Arming of g-switch: Launch-ready.

### 7.8.9.2 Test (Flight) Operations

Upon launch, the g-switch activates the timer which, in turn, activates and de-activates each experiment module in sequence and changes the power level of each experiment according to a preset program. The power program for each experiment is provided by preset controls which are integrated in the inverter unit.

### 7.8.9.3 Post-Test (Ground) Operations

- (31) Depressurization of expt. chambers.
- (32) Recovery of samples.

7.8.9.4 Time Diagrams. The sequence of experiment phases and events for rocket experiments is identified in the detailed time diagram, Fig. 7.8-4

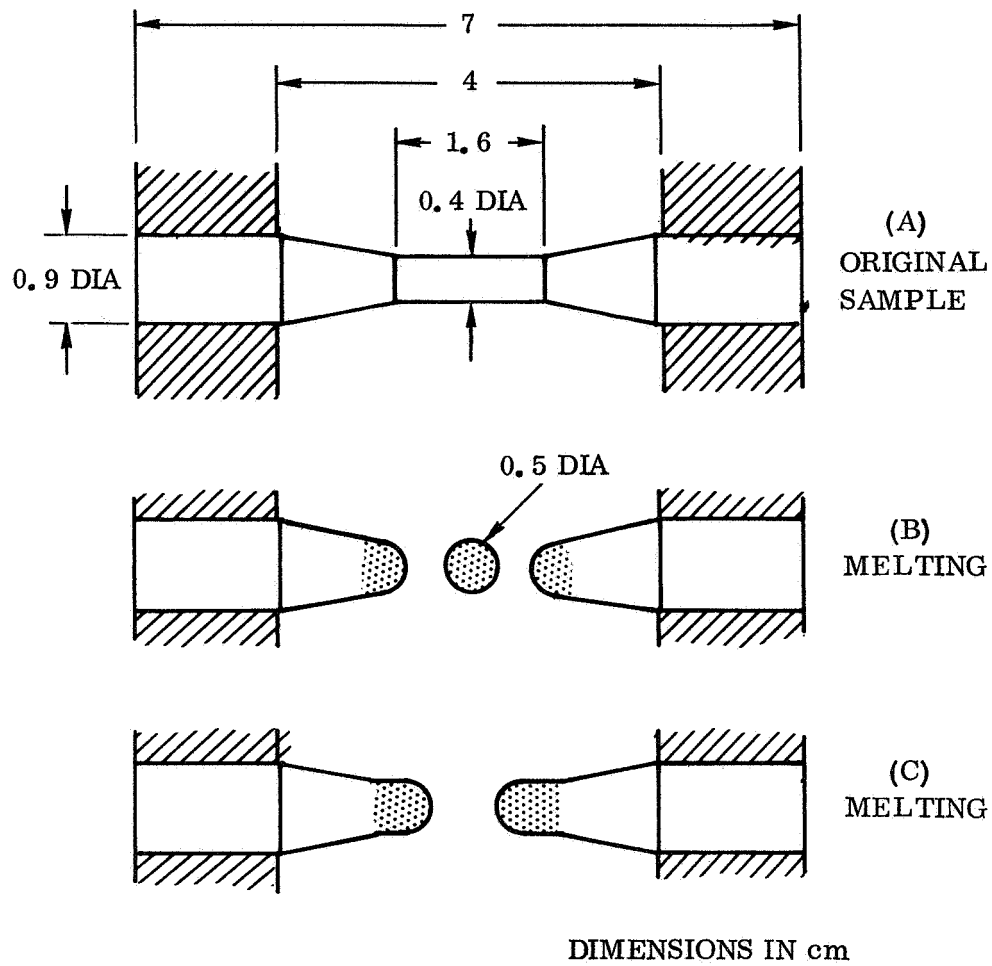


Figure 7.8-1. Sample Configurations for Superconductor Experiments

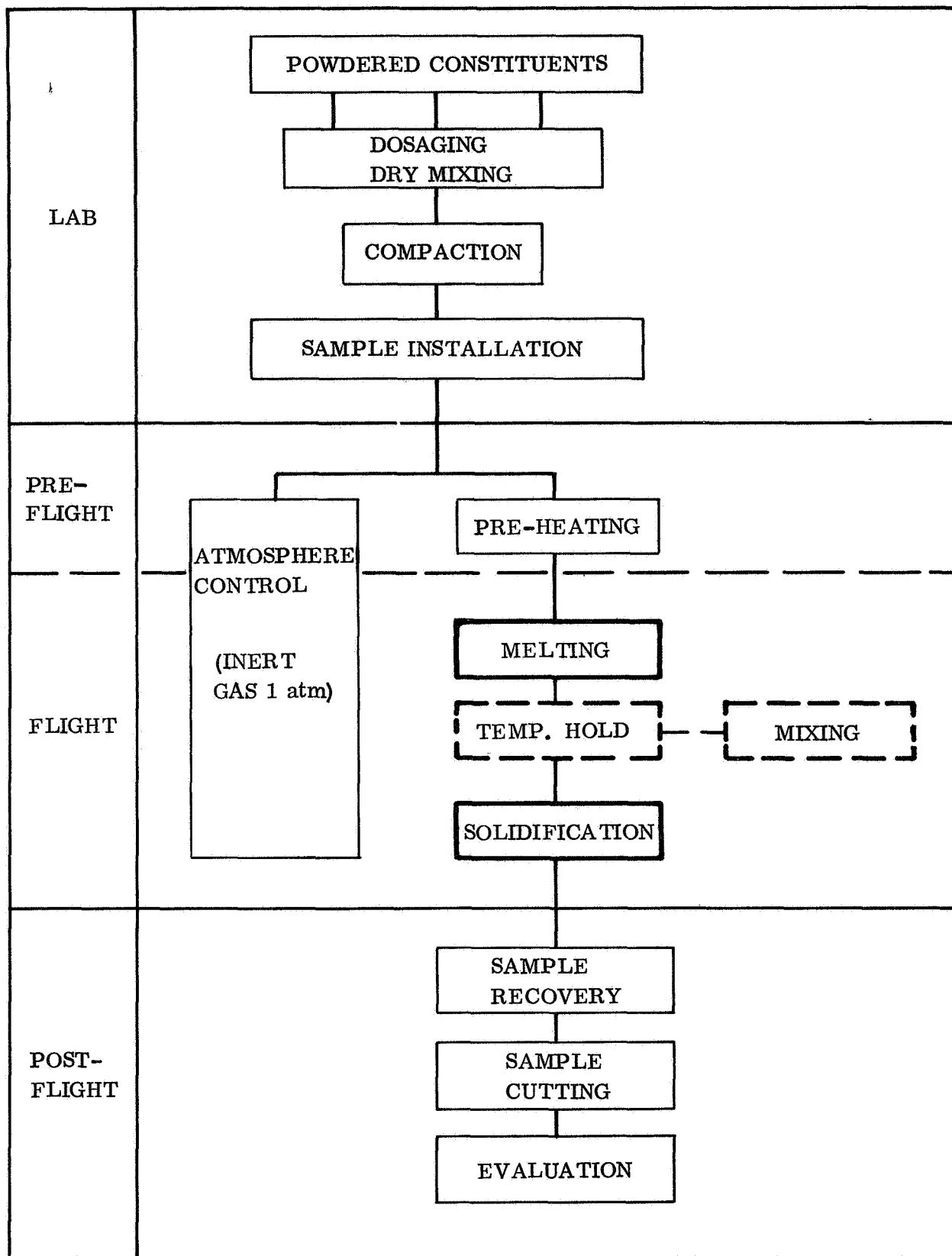
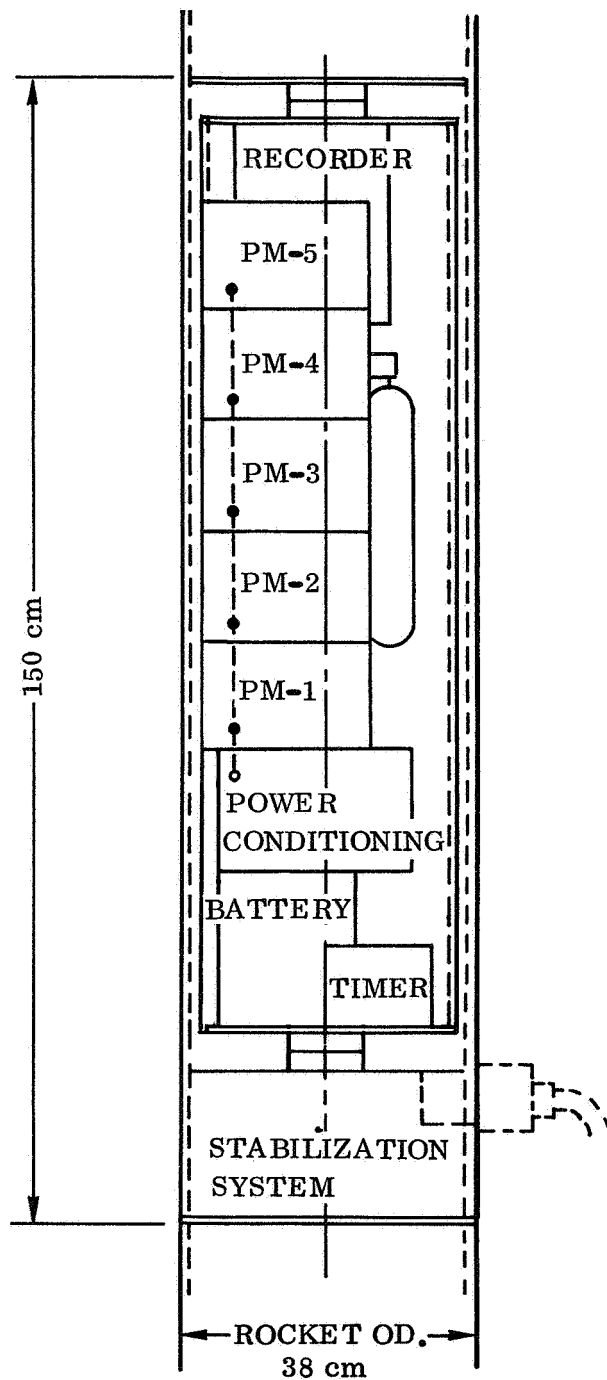


Figure 7.8-2. Experiment Flow Diagram — Superconductors

# ROCKET PAYLOAD ASSEMBLY



PM = PROCESSING MODULES  
ALL SAMPLES IN ROCKET AXIS  
PAYLOAD WEIGHT 127 Kg

Figure 7.8-3. Rocket Payload Assembly for Superconductor and Metastable Alloy (Level II) Experiments



# EXPERIMENT PROGRAM – TRAJECTORY B

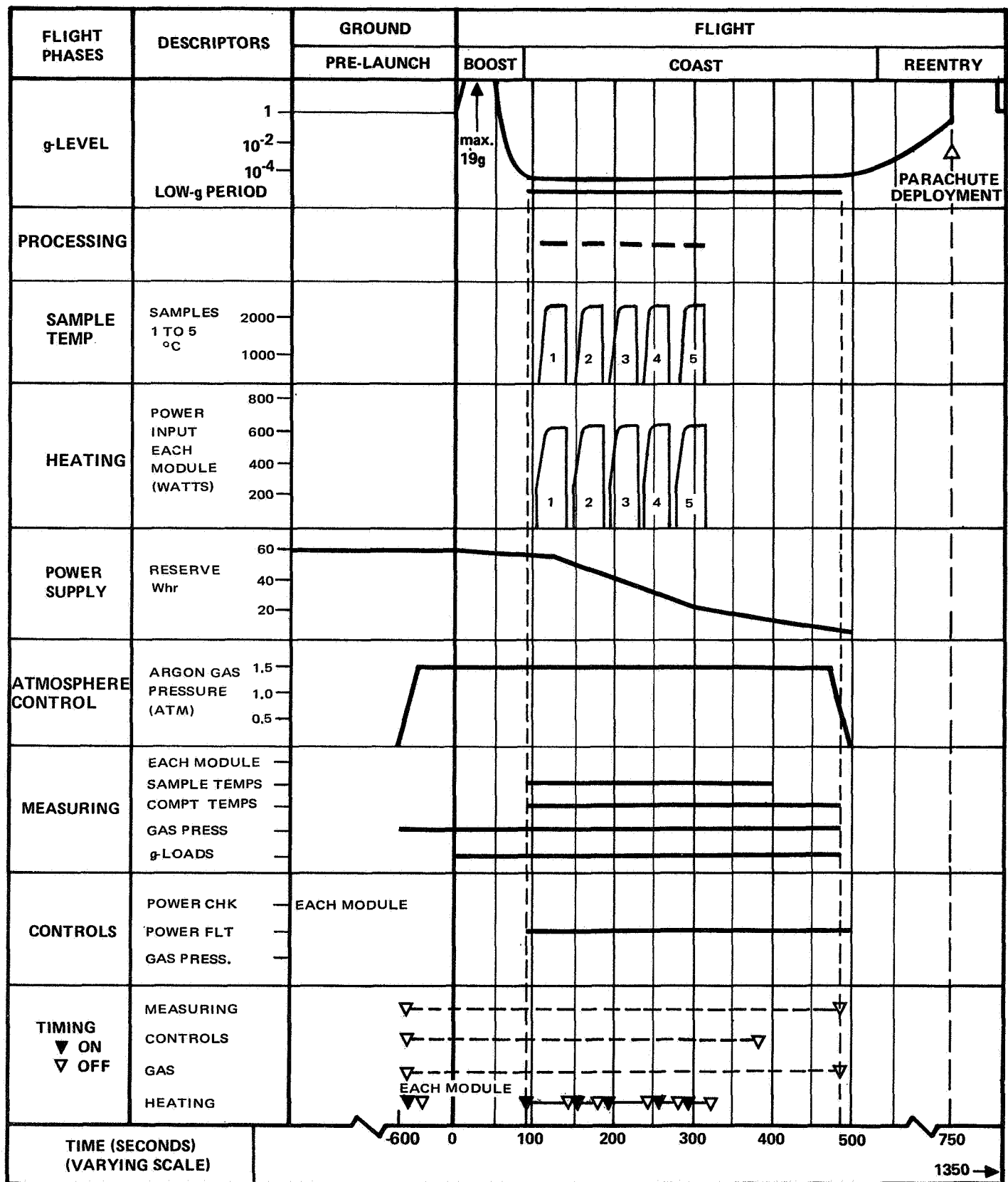


Figure 7.8-4. Time Diagram for Superconductor Rocket Experiments (Level II)

## 7.9 METASTABLE ALLOYS (IMMISCIBLES) - THERMAL DISPERSION - MODERATE TEMPERATURE

### 7.9.1 Concept and Objectives

In a sizeable number of alloy systems, and/or composition ranges thereof, complete miscibility is only attained above a discrete temperature between the melting and the boiling point (consolute temperature). The terrestrial preparation of alloys from such material systems is impossible, since they exhibit immiscibility in the liquid state prior to solidification, resulting in gravity-induced segregation of alloying elements. In the absence of gravity and segregation, the liquid components co-exist in a stable liquid dispersion, which may be retained through solidification down to room temperature. The resulting solid material is essentially an ultra-fine composite which may exhibit alloy characteristics. From the 300 identified immiscible material systems, new families of "alloys" may emerge which cannot be obtained terrestrially. Promising applications of these materials are indicated as semiconductors, superconductors and as catalysts.

The concept of producing metastable alloys from immiscible elements has been proven in experiments on the Apollo 14 mission and in low-g tests in the MSFC drop tower and in the KC-135 research aircraft.

It is apparent that the beneficial characteristics of the end-product material depend on the fineness of dispersion and, consequently, the process parameters responsible for dispersion, thermal dispersion and homogenization (mechanical dispersion). From the viewpoint of test requirements, a distinction is further made as to maximum processing temperature, identifying two regimes: (a) low and moderately high temperatures with a limit of 1000° C and (b) processing temperatures above 1000° C. Experimental processes are, therefore, divided into four categories:

- (a) Thermal Dispersion - Max. Temp. 1000° C
- (b) Thermal Dispersion - Processing Temp. above 1000° C
- (c) Homogenization - Max. Temp. 1000° C
- (d) Homogenization - Processing Temp above 1000° C

This section is concerned with process (a), processes (b) and (c) are evaluated in the two subsequent sections 7.10 and 7.11. Process (d) is excluded, since the leadtimes for the development of high temperature homogenization techniques exceed the time-frame of the initial low-g test program.

Thermal dispersion is achieved by the following treatment: (1) heating through melting to a temperature level above the consolute temperature; (2) temperature hold at this level to effect complete solution; and (3) cooling through solidification. Dispersion is generated in the cooling period (3) through the immiscible liquid regime between the consolute temperature and solidification. During this period zero-g or low-g levels have to be maintained to prevent segregation or coagulation of constituents, while phases (1) and (2) are not g-sensitive.

The degree and homogeneity of dispersion is determined by three factors: (a) complete solution of constituents or adequate length of temperature hold during phase (2); (b) lowest possible g-level during phase (3) and (c) slow cooling rate during phase (3).

From the viewpoint of low-g experiment requirements, postulation (a) is no problem since it is not g-sensitive and can be accomplished prior to the low-g test period. This leaves only the g-level and the cooling rate as criteria for the degree of dispersion. Since both criteria are related to the basic limitations of low-g test facilities (g-level and time at this level). Two verification levels are introduced:

Verification Level I: Intermediate g-level in the order of  $10^{-2}$  g and high cooling rate (solidification within seconds). This verification level is adequate for exploratory experiments and small masses of material.

Verification Level II. Low g-level in the order of  $10^{-4}$  g, slower cooling rate and/or larger material masses. This verification level permits a more accurate determination of the achieved material properties.

In both cases, the end product characteristics are verified exclusively by laboratory-evaluation of the processed material sample, and no property measurements are required during experiment performance.

### 7.9.2 Verification Requirements

Verification of process parameters and obtained material characteristics calls for the following:

- (1) Experimental conditions which facilitate cooling rates in the order of  $10^3$ ° C/sec for verification level I and 5-20° C/sec for verification level II.
- (2) Microscopic material evaluation for particle size, particle distribution (homogeneity of dispersion and bulk material characteristics, such as porosity or voids).
- (3) Electrical measurements, including resistivity and/or inductance, Hall coefficient and Hall mobility.
- (4) Adequate sample size for (2) and (3).

For some materials, an evaluation is further indicated for potential application as catalysts which involves the determination of particle number and surface area per bulk material surface unit.

### 7.9.3 Materials

Thermal dispersion is primarily aimed at verification level II with slow cooling rates, while preliminary experiments at higher cooling rates (verification level I) may serve for the determination of exact composition and processing parameters for verification level II.

For low cooling rates, compositions of lower melting and processing temperatures are preferred since they minimize the problems of temperature control and container material. Upon careful review of candidate compositions with J. Reger of TRW, the following experimental materials have been selected; the listing identifies the initial compositions in mol ratios, the consolute temperature (min. processing temperature to achieve solution), the temperature at which all constituents are molten, and the lowest temperature for solidification.

<u>Alloy System</u>	<u>Composition (Mol Ratio)</u>	<u>Consulate Temp ° C</u>	<u>Melting Temp ° C</u>	<u>Solidif. Temp ° C</u>
Al-In	AlIn <sub>3</sub> AlIn	875	660	157
Ca-La	CaLa	900	920	842
Cu-Pb	Cu <sub>3</sub> Pb CuPb <sub>3</sub>	990	1083	328
Ga-Pb	Ga <sub>3</sub> Pb GaPb <sub>3</sub>	606	328	29

#### 7.9.4 Material Quantity and Sample Size

The material quantity is dictated by the minimum sample size required for the measurement of electrical properties (3) in 7.9.2. For thermal dispersion, a sample of 1 cm length and 0.25 cm<sup>2</sup> cross section is considered as adequate for both verification levels. However, for verification level II a larger mass in the order of 2 cm<sup>3</sup> is desirable to evaluate size effects and to accommodate the preparation of several evaluation samples. The following sample sizes, material quantities and weights have, therefore, been selected for low-g experiments.

<u>Sample</u>	<u>Verification Level</u>	
	<u>I</u>	<u>II</u>
Configuration (cm)	1 x 0.5 x 0.5	2 x 2 x 0.5
Mat. Quantity (cm <sup>3</sup> )	0.25	2
Weight (gr) AlIn	1.25	10.0
AlIn <sub>3</sub>	1.54	12.3
CaLa	0.97	7.74
CuPb	2.5	20.2
CuPb <sub>3</sub>	2.7	21.4
Ga <sub>3</sub> Pb	1.81	14.5
GaPb <sub>3</sub>	2.49	19.9

Samples for verification levels I and II are further referred to as samples I and II.

Both samples require a thin (for good heat transfer and thermal expansion) container of equal size. Container material should be wetted inside by at least one alloy component and should be treated outside for high emissivity.

#### 7.9.5 Processing Phases

A complete experiment consists of the following major process phases (\* = g-sensitive):

Sample preparation and installation

Heating to processing temperature

Temperature hold at processing temperature

\*Cooling to complete solidification

Sample recovery and evaluation.

The sequence of individual processing steps is identified in the flow diagram, Fig. 7.9-1.

Bold frames indicate g-sensitive phases.

#### 7.9.6 Low-g Test Requirements

##### 7.9.6.1 Experiment Time Requirements

(1) Heating: The time for heating to the liquid state processing temperature is listed below for samples I and II. It is based on the material system with the highest heating requirements and a radiation heater with a filament temperature of app. 1300° C.

(2) Temperature Hold: The minimum "hold" time at the processing temperature is independent of sample size. The time listed below applies for the conditions stated in (1). Both, phases (1) and (2) are not g-sensitive; they are therefore preferably carried out prior to low-g test performance by ground pre-heating and continued to the cooling start time in the low-g period. The pre-test heating time can be extended, if necessary.

(3) Cooling: The cooling time below is defined for two time periods: the low-g period and not-g sensitive period. The data for verification level I are based on turbulent water cooling in direct contact with the sample container. The critical g-sensitive

period is the cooling from the processing temperature to complete solidification (solidification of the lowest-melting constituent). Indications are that the degree of dispersion and the resulting alloy quality are inversely related to the cooling rate or, in other words, increase with the length of the cooling period. This is based on circumstantial evidence, derived from drop tower and KC-135 experiments. In the absence of extended low-g data, it can only be postulated that the cooling period should be as long as possible. For extended-time low-g experiments, the cooling phase has to be necessarily divided into two periods: A passive and an active period. The time for final solidification by active cooling is in the order of 60 seconds, regardless of total cooling time. The minimum time for the passive period has been placed rather arbitrarily at 100 seconds as an order-of-magnitude-step from tower and KC-135 experiments (2-10 sec.). However, longer periods are desirable.

(4) The Time Requirements for level I and II experiments are summarized below.

<u>Verification Level/Sample</u>	<u>I</u>	<u>II</u>
<u>Individual Phases (Seconds) (* = g-sensitive periods)</u>		
Melting	300	60
Hold at Max Temp	900	900
*Low-g Hold at Max Temp	0.5	-
*Passive Cooling	-	>100
*Active Cooling	2.5	60
Terminal Cooling	300	600
<u>Total Times (Seconds)</u>		
Preheating (1-19g)	2100	1000
*Low-g Processing	3	>160
Terminal Cooling	300	600

7.9.6.2 Heat and Power Requirements. The max net heat required to bring the sample materials (only) to the processing temperature is app. 650 cal, for sample I and 3600 cal

for sample II. The related energy input depends extensively on the type of heating device. For level I experiments, there is a considerable latitude in power consumption and, consequently, the choice of heating method. For level II experiments and the processing device identified in 7.9.8 the power requirements are as follows:

	<u>Time (sec)</u>	<u>Max Rate (w)</u>	<u>Energy (wh)</u>
Heating to 1000° C	60	1800	25
Hold at 1000° C	900	650	162
Active Cooling	600	100	17
Support Systems	1500	150	65

Only a fraction of these requirements apply to low-g conditions, as further detailed in Sect. 7.9.7.2.

7.9.6.3 g-Level. The max g-level during the passive and the initial active cooling periods should not exceed  $10^{-4}$  g (desirable) or  $10^{-3}$  g (acceptable).

#### 7.9.7 Low-g Facilities and Experiments

A comparison of the required low-g times, above, and low-g facility capabilities, Section 4.0 shows that Type I experiments can be conveniently carried out in the MSFC 300-foot drop tower, and Type II experiments in research rockets.

7.9.7.1 Drop Tower Experiments. For level I tower experiments, a single experiment per drop is preferred. All heating is carried out under one-g and stopped at the moment of vehicle release. Active cooling starts 1/2 second after release and is continued for 300 seconds to preclude remelting of constituents. The weight, space and power requirements of the apparatus are well within the capabilities of the drop tower package (Section 4.1).

7.9.7.2 Rocket Experiments - Facility Selection. The required low-g time, comprising passive and active cooling phases, has been defined in Section 7.9.6.1 as 160 minimum, with the stipulation that longer times are highly desirable. Therefore, two facilities,



representing two time capabilities are identified; the ultimate selection depends on programming and cost considerations.

<u>Verification Level</u>	<u>IIa</u>	<u>IIb</u>
Facility	RR-1	RR-4
Trajectory	A	B
Low-g Time (sec)	243	390
Payload Weight (kg)	130	125

7.9.7.3 The Max. Number of Experiments/Flight is determined by payload weight, space and power requirements.

<u>Payload Weight</u>	
RR-1/Traj. A Capability	130 kg
RR-4/Traj. B Capability	125 kg
4-Expt. Payload	117 kg
Margin	8-13 kg

<u>Payload Space (in Axial Height)</u>	
Net Space in Support Module	80 cm
4 Expt. Modules	72 cm
Argon System	6 cm
Total 4-Expt. Space	78 cm
Margin	2 cm

#### Power/Time Requirements

Since the heating/melting phases are not g-sensitive, experiments can be scheduled so that most of the heating energy is provided by ground power. The resulting reduction of the flight power rates further permits to schedule all flight processing phases concurrently. The optimum processing and power schedule for experiments IIa (RR-1-A) and IIb (RR-4-B) are detailed below; in both cases, four experiments can be conveniently accommodated with regard to low-g time, max. power rate and total power consumption.

<u>Count-Down</u> <u>Time (sec)</u>	<u>Processing</u> <u>Phase</u>	<u>Energy Rate</u> <u>1 Expt. (w)</u>	<u>Energy Rate</u> <u>Payload (w)</u>	<u>Energy Con-</u> <u>sumpt.(wh)</u>
----------------------------------------	-----------------------------------	------------------------------------------	------------------------------------------	-----------------------------------------

Experiments IIa (RR-1/Trajectory A)

-1050 to -810	Heating to 1000° C	(1000)	(1150-3100)	-
-810 to 0	Hold at 1000° C	(650)	(2750)	-
0 to 90	Hold at 1000° C	650	2750	69
90 to 270	Passive Cooling	-0-	150	8
270 to 330	Active Cooling	100	550	9
330 to 930	Term. Cooling	100	550	92

Experiments IIb (RR-4/Trajectory 3)

-870 to +90	Same as above)			69
90 to 420				15
420 to 480				9
480 to 1080				92

\*)Including 150W continuous for support (recorder etc.) Data in ( ) = ground supplied power.

Power Assessment

Battery Supply	220 wh
Flight Requirements IIa	178 wh
Flight Requirements IIb	185 wh
Margin	35-42 wh

7.9.7.4 Number of Samples. The chamber of the furnace module defined in 7.9.8.2 can easily accommodate 2 samples (7.9.4) in tandem with a dimensional envelope of 4 x 2 x 0.5 cm. The two samples have to be compatible with the same processing temperature and represent preferably two compositions of one alloy system, such as  $\text{Cu}_3\text{Pb}$  and  $\text{CuPb}_3$ . The use of 2 samples has no significant effect on the defined processing conditions and requirements with the exception of the heat-up time to the max temperature; this is, however, immaterial since it is a ground operation.

7.9.7.5 Experiment Definition. The results of this assessment are summarized as follows:

Verification Level	I	IIa	IIb
Low-g Facility	MSFC Tower	RR-1	RR-2
Trajectory	-	A	B
Expts. per drop/flight	1	4	4
Samples per drop/flight	1	4-8	4-8
Low-g Processing Time (sec)	3	240	390
Total Processing Time (sec)	1,500	1,800	1,950
Max. Sample Temp. (°C)	1,000	1,000	1,000

#### 7.9.8.1 Drop Tower Payload Assembly

For drop tower experiments the apparatus design developed by TRW and used successfully in initial tower experiments has been adopted. Major components and their approximate weight are:

Processing Chamber	1	kg
Water Supply	15	kg
Gas Supply	10	kg
Valves	6	kg
Sequencer	10	kg
Structures	40	kg
Wiring, Plumbing	3	kg
<hr/> Complete Assembly	85	kg

A cross section of the TRW processing unit is shown in Fig. 7.9-2. One complete assembly is used per experiment (one sample per drop). The support module is the standard MSFC drop package assembly.

7.9.8.2 Rocket Processing Module. The design of the rocket processing module is determined by two criteria, peculiar to the defined experiments:

- (a) "Hold" time in the order of 10-15 minutes at the max. processing temperature of 1000° C. This period is, however, not g-sensitive and can be into the pre-launch and boost phases.
- (b) Terminal cooling to a constant temperature below 140° C, to assure solidification of the material component of lowest melting temperature (indium).

Both furnaces described in Section 6.2.1 are acceptable; preference is given to the processing module illustrated in Fig. 6-3 and defined in Section 6.2.1.1, since it has the highest coolant volume and heat capacity. To be on the safe side, it is desirable that the heat capacity of the cooling system is substantially higher than the total heat stored in the module prior to terminal cooling. Disregarding the small fraction of the total energy transferred to the payload section environment, the energy balance and the resulting terminal temperature for the individual module and a max. temperature of 1000° C are as follows:

Heat Stored in Module	147,000	cal
Coolant Capacity	3.7	liters
Max Heat Absorption Capacity (Max Coolant Temp. 90° C)	185,000	cal
Excess Coolant Capacity	38,000	cal
Terminal Coolant Temperature	80	° C

These data represent extreme conditions (1000° C); for most experiments (sample materials), the max. temperature is lower, resulting in a higher coolant capacity excess and a lower terminal temperature.

The configuration of the processing module is illustrated in Fig. 6-3. Major data are as follows:

Diameter	28 cm
(Axial) Height	18 cm
Chamber Volume	200 cm <sup>3</sup>
Max. Power Rating	1800 watts
Weight (with coolant)	14 kg

Argon supply is required only for experiments in excess of 900° C (oxidation control of heating element); it may be eliminated by replacement of the heating element after each check-out and flight test.

7.9.8.3 Rocket Support Module. The outfitting and weight of the support module are (numbers in parenthesis identify components specified in Section 5.2.2 and Table 5 - 1):

Basic Hardware (1, 2, 3)	33 kg
2 Batteries (4)	6 kg
Power Conditioning (5A, B, C-2)	12 kg
Sequencer, Recorder (6, 7)	4 kg
Contingency (8)	2 kg
	<hr/>
Total Weight	57 kg
Net Space Available for Processing Modules (axial Height)	80 cm
Total Power Supply	220 wh

7.9.8.4 Rocket Payload Assembly. The payload assembly is illustrated in Fig. 7.9-3. It consists of four processing modules, the support module and an optional argon system. Major payload data are:

Payload Weight

4 Processing Modules	56 kg
Support Module	57 kg
Argon Supply	4 kg
Total Payload	117 kg
Rocket 1-A Capacity	130 kg
Reserve	13 kg

Payload Dimensions

Max. Diameter	38 cm
Apparatus Diameter	28 cm
Apparatus Height	78 cm
Height Margin	2 cm

## Payload Power

Max. Preheating Rate	1800 watts
Stored Board Energy	220 wh
Flight Consumption	185 wh
Flight Power Contingency	35 wh

### 7.9.9 Experiment Performance

The sequence of operations is essentially the same for tower and rocket experiments. For simplicity, the terminology of rocket experiments is used.

7.9.9.1 Ground Operations. After check-out of all systems, experiment performance starts 16 minutes before launch. Ground operations comprise:

- (1) Activation of Board Recorder
- (2) Pressurization of processing chambers (if applicable)
- (3) Sequential heating of each module to max. temperature from ground power
- (4) Hold at max. temp. to launch.
- (5) Status check
- (6) Arming of g-switch

7.9.9.2 Flight Operations. At launch power is switched over to payload battery.

Heating and recording continues uninterrupted. Flight operations are:

- (1) Activation of timer by g-switch
- (2) Continued heating (temp. hold)
- (3) Radiation cooling (passive)
- (4) Active cooling (water quench)
- (5) Deactivation of all systems.

The detailed sequence of operations and events is identified in the time diagram, Fig. 7.9-4.

### 9.9.3 Post-Flight Operations

- (1) Payload recovery
- (2) Recovery of samples
- (3) Check consumption of expendables.

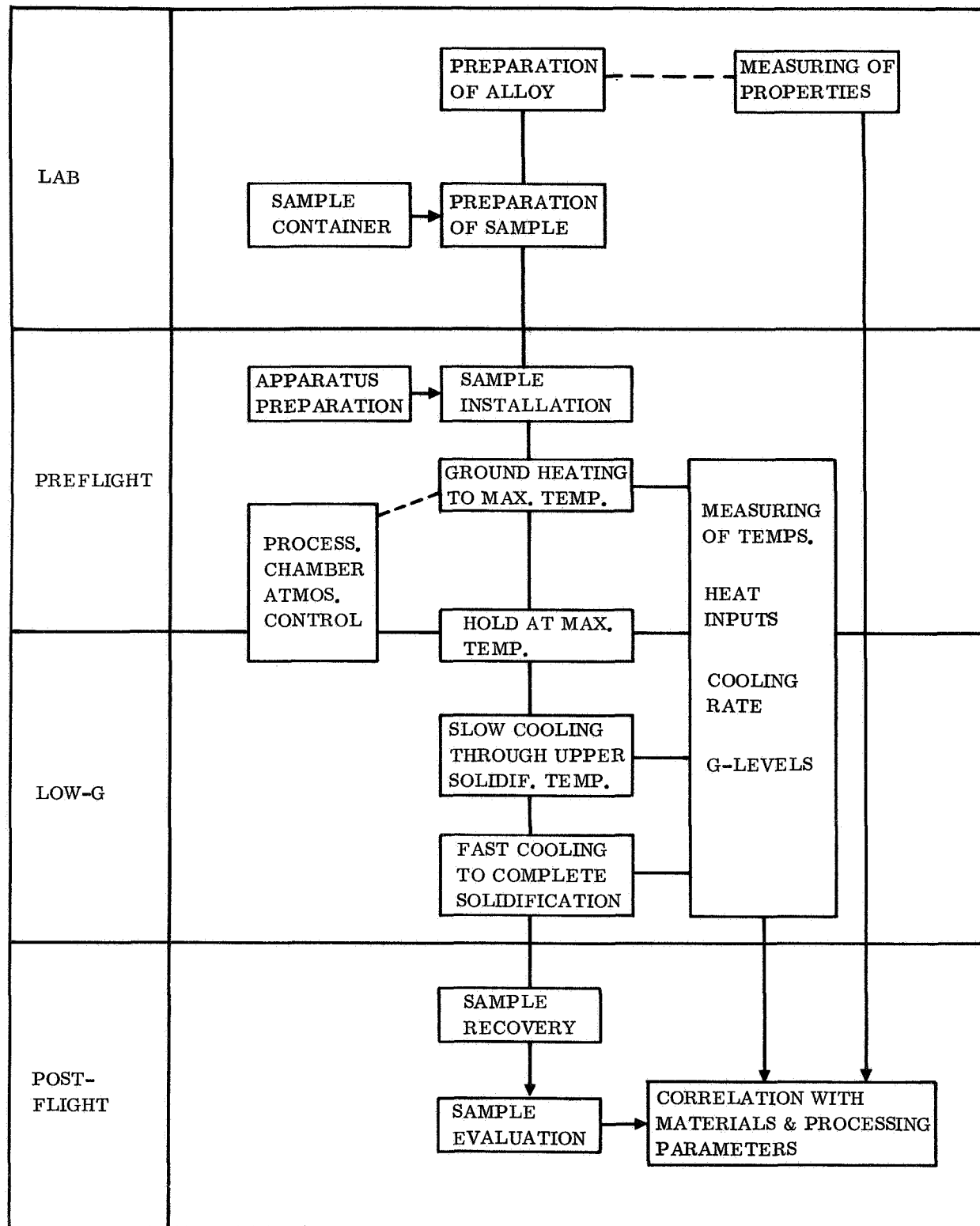


Figure 7.9-1. Process Flow Diagram

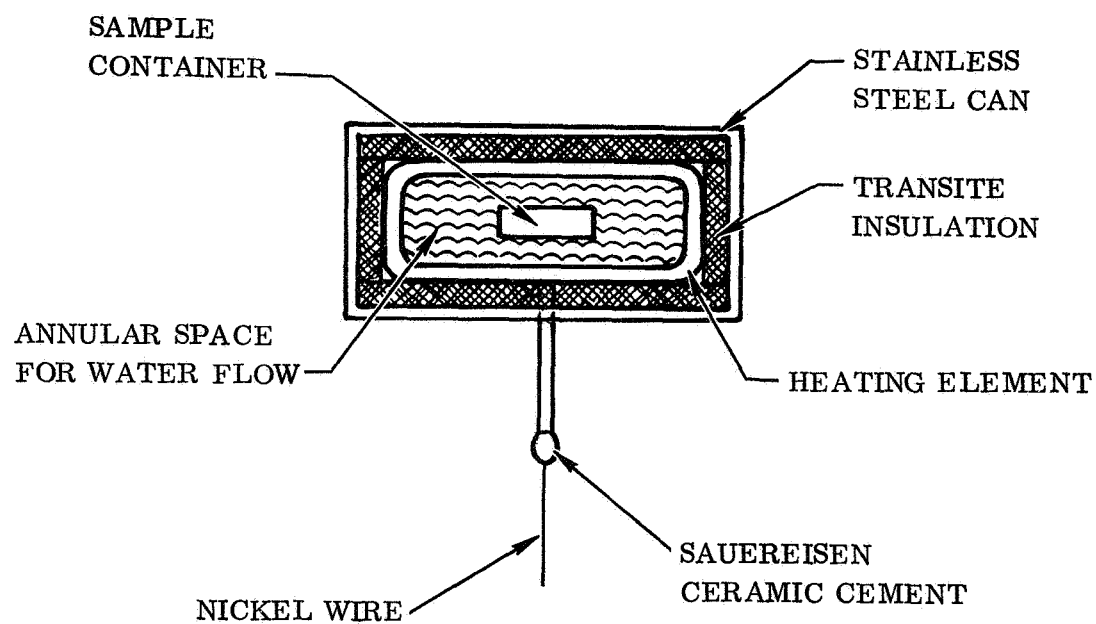


Figure 7.9-2. Processing Chamber for Drop-Tower Experiments - Metastable Alloys (TRW)



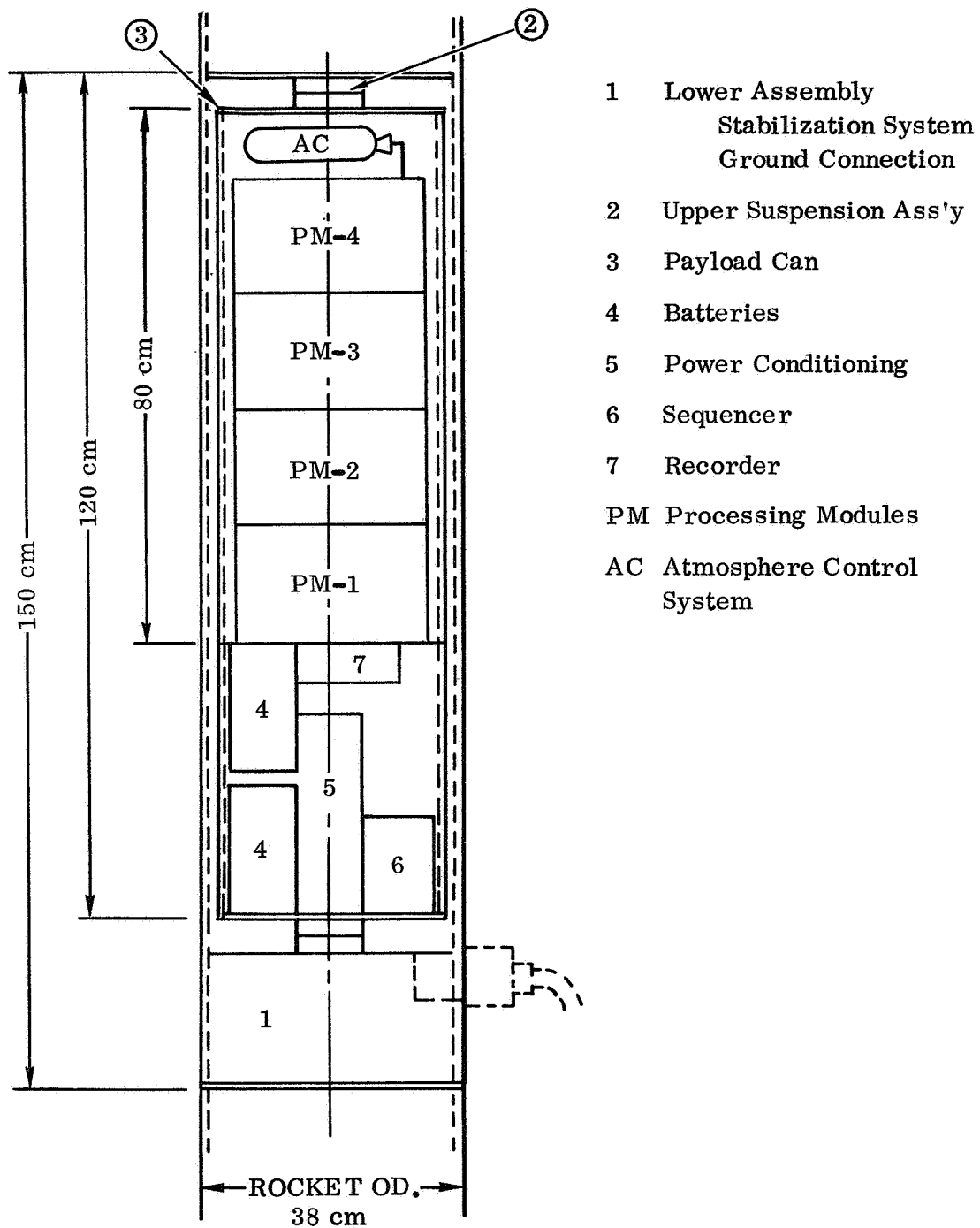


Figure 7.9-3. Rocket Payload Assembly

# EXPERIMENT PROGRAM – TRAJECTORY A

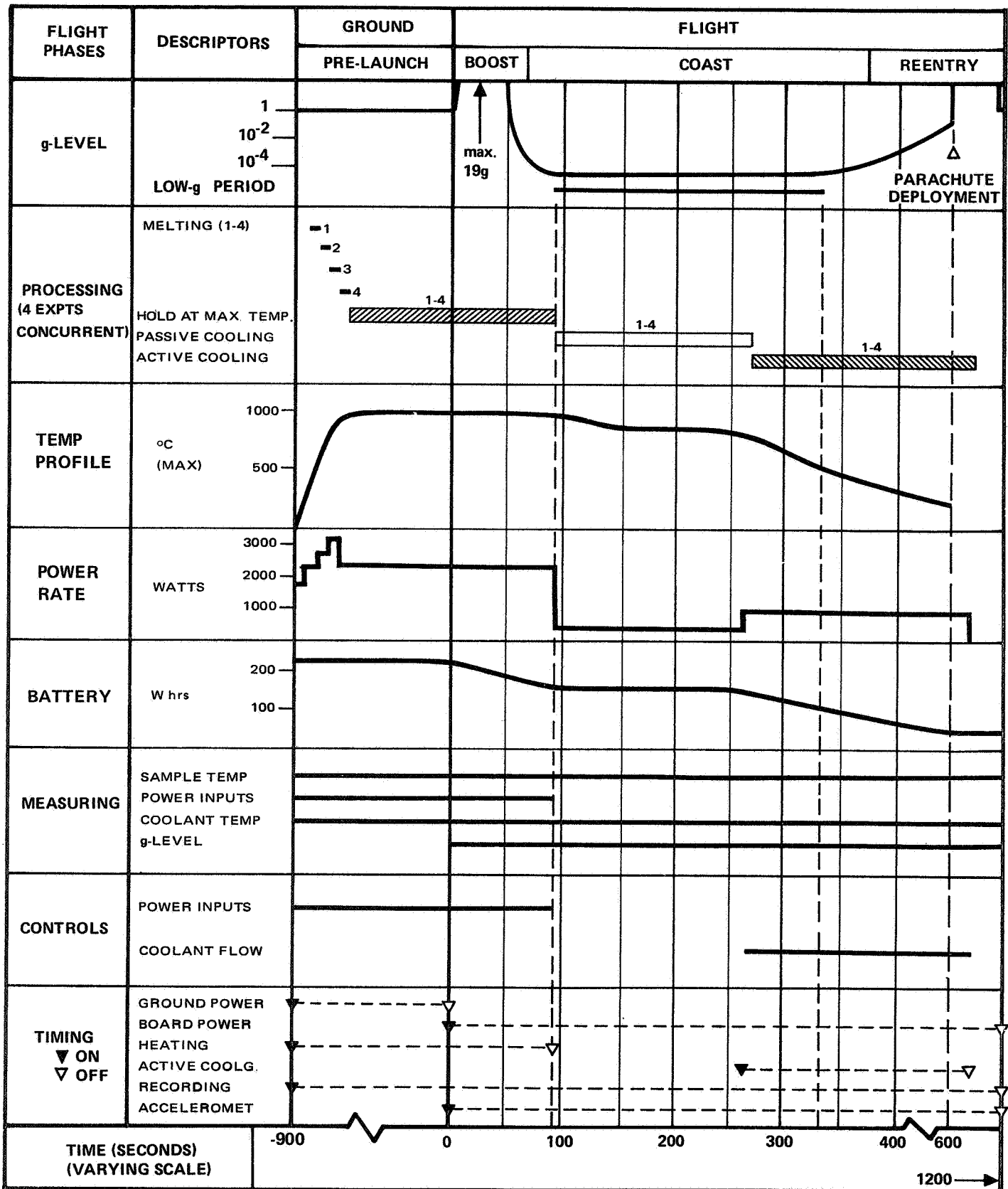


Figure 7.9-4. Time Diagram - Rocket Experiments

## 7.10 METASTABLE ALLOYS - THERMAL DISPERSION - HIGH TEMPERATURES

This process comprises the preparation of metastable alloys (immiscibles) with processing temperatures above 1200° C by thermal dispersion.

### 7.10.1 Process Definition and Objectives

(Refer to Sect. 7.9.1)

### 7.10.2 Verification Requirements

Verification requirements are identical to those defined in Sect. 7.9.2, except for the cooling rate. The high temperatures involved in this process group permit reduced hold periods at the consolute temperature, since the high thermal activation enhances the establishment of complete solution. The high temperatures further increase the passive cooling rate, which has a positive and a negative aspect. On the positive side, this eliminates the need for active terminal cooling, since complete solidification, including the lower-melting alloy component, can be achieved by radiation. On the negative side, a high cooling rate may affect the quality of dispersion. Since the control of the cooling rate is a matter of experiment complexity, a choice of two experimental techniques is introduced, represented by the following two verification levels:

Verification Level I: Short time at the consolute temperature and high cooling rate - cooling temperature profile as resulting from radiant heat dissipation of the bare sample. The time to solidification of the high-melting constituent is app. 1 second, and 5-20 seconds for the low-melting constituent.

Verification Level II: Extended time at the consolute temperature and reduced cooling rate. The time to solidification of the high-melting constituent is app. 3-5 seconds, and 15-60 seconds for the low melting constituent.

### 7.10.3 Experimental Materials

Candidate alloys which have been selected<sup>\*)</sup> for these experiments, in the order of decreasing consolute (max. processing) temperatures are:

<u>Alloy</u>	<u>Max. Processing Temp. (° C)</u>	<u>Density (g/cc)</u>
Nb-La <sub>3</sub>	2500	6.8
Nb <sub>3</sub> -La	2500	8.0
Ta-La <sub>3</sub>	3000	8.8
Ta <sub>3</sub> -La	3000	14.0
Ta <sub>3</sub> -Y	3000	13.6
Cr-(Cr <sub>2</sub> O <sub>3</sub> )	2300	6.1
Fe-(FeO)	1600	6.8
Ge-(GeO)	1250	6.0

---

\*) Alloy selection based on consultation with J. Reger of TRW.

The Nb<sub>3</sub>-La system is used as model for the experiment evaluation, since it represents a fairly high processing temperature. It furthermore exhibits the highest heat content at processing temperature (liquid solution) of 300 cal/gr (Ta<sub>3</sub>La = 190 cal/gr; Ta<sub>3</sub>Y = 265 cal/gr).

#### 7.10.4 Material Quantity and Sample Configuration

7.10.4.1. Sample Size and Heating Method. The minimum sample size is dictated by the measurement of electrical properties (7.9.2). Adequate measurements can be obtained with a sample of 0.4 cm diam x 0.4 cm; increased sample length enhances the accuracy of measurements and the choice of evaluation technique. The ideal heating method for this relatively small sample size in combination with the high processing temperatures is direct resistance heating, as discussed in Section 6.2.3.

7.10.4.2 Sample Configuration. The standard resistance heating sample, Fig. 6-13a, satisfies the min. sample size requirements perfectly. For verification level I, a bare sample is used, whose melting patterns are illustrated in Fig. 6-13b and c. For verification level II, a (split) ceramic sleeve is placed over the sample, as illustrated

in Fig. 7.10-1. The prime purpose of this sleeve is to maintain the cylindrical sample configuration throughout the low-g melting cycle. It further reduces the passive cooling rate, extending the solidification time. It provides the requirements for verification level II as defined in 7.10.2 with regard to both, sample size and cooling rate. The increase of power requirements for the heating of the enclosed sample is insignificant as the heat content of the sleeve is offset by its insulating effect upon the sample material.

7.10.4.3 Material Quantities. The original sample configuration and, consequently, material volume is identical for both sample types. The data for the sample (processed specimen section) and the complete specimen (including contact ends) are:

<u>Dimensions</u>	<u>Sample</u>	<u>Specimen</u>
Diameter (cm)	0.4	0.7 (max)
Length	1.6	6.0
Volume	0.2	1.6
<u>Weights (gr)</u>		
Model ( $\text{Nb}_3\text{-La}$ )	1.6	12.8
Max. ( $\text{Ta}_3\text{-La}$ )	2.8	22.4
Min. ( $\text{Ge - GeO}$ )	1.2	9.6

After low-g testing, the final sample sizes and material quantities available for evaluation measurements are:

	<u>Configuration</u>	<u>Dimens. (cm)</u>	<u>Vol (cm<sup>3</sup>)</u>
Sample I	1 Sphere	0.45 diam	0.047
	<u>plus</u>	(each)	(each)
	2 Half-spheres	0.55 diam x 0.3	0.045
Sample II	Cylindrical	0.45 diam x 1.6	0.20

Sections 7.10.5 through 7.10.9: For Verification Level I all requirements are identical to 7.8 (superconductors). The following evaluation is, therefore, confined to Level II.

#### 7.10.5 Experimental Process Definition

Level II experiments (extended time at the consolute temperature) comprise the following major processing phases:

1. Sample preparation
2. Sample processing
  - a. Heating through melting to the consolute temperature
  - b. Hold at the consolute (max) temperature for 30-60 seconds
  - c. Passive/radiation/cooling to complete solidification, including the lowest-melting constituent).
3. Sample evaluation

Individual processing steps are identified in the process flow diagram, Fig. 7.10-2 . Bold frames identify g-sensitive phases.

#### 7.10.6 Low-g Test Requirements

The g-sensitive process period extends from the start of melting to completed solidification.

7.10.6.1 Heating Method: Direct resistance heating, enclosed sample, as defined in 7.10.4.

7.10.6.2 Low-g Time Requirements. Even though a slow heating rate during phase 2a, above, may be desirable, its contribution to effective solution is of secondary significance and high heating rates, in order to conserve low-g time, are acceptable for the selected resistance heating method; at a reasonable power input, the time of the heating/melting phase is in the order of 30 seconds. Fig. 7.10-3 shows a typical heating profile.

The times at consolute temperature (2b) and for solidification (2c) should be as long as possible. There a compromise has to be accepted between the desirable time and reasonable low-g time requirements. It should be noted, that even at the short times of tower and KC-135 experiments in the order of a few seconds, metastable alloys were produced successfully. A increase of the time by a factor of 10-20 is expected to bring

out all metallurgical effects and generate product properties which represent at least 90% of those attainable under ideal conditions. The time requirements for phases 2b and 2c have, therefore, been placed at 30 seconds for each phase. The low-g time requirements are summarized as follows:

Heating to max. temp (2a)	30 sec
Hold at consolute temp (2b)	30 sec
Solidification (2c)	30 sec
Min total low-g time	90 sec.

#### 7.10.7 Low-g Facilities and Experiments

The total required low-g time of 90 seconds can be conveniently provided by rocket class 1 or 2 and trajectory A (standard WSMR). The number of experiments which can be carried out on one flight is determined primarily by power limitations.

7.10.7.1 Number of Experiments/Flight. From the viewpoint of payload weight and space limitations, 6 experiments can be accommodated, as defined in 7.8 (Superconductors) representing identical equipment. It has been found that 6 experiments are also feasible with regard to power/time limitations within the total low g-time of 243 seconds by the sequential scheduling of 2 groups of experiments, each consisting of 3 concurrent experiments. In this arrangement it was possible to increase the times for phase 2b and 2c from the required min. of 30 seconds to 50 seconds, each. The extension of the cooling time called for the reduction of the high radiative cooling rate during the first 25 seconds by continued and controlled heating (programmed power input decreasing from 600 to 0 watts). The optimized experiment schedule is as follows:

<u>Low-g Time</u> (Sec)	<u>Expt. 1, 2, 3</u>	<u>Expts. 4, 5, 6</u>	<u>Total Power Rate</u> (W)
0-30	Melting		2400
30-80	Max. temp hold		2400
80-105	Contr. cooling		1500
105-135	Passive cooling	Melting	2400
135-185		Max. temp. hold	2400
185-210		Contr. cooling	1500
210-240		Passive cooling	150

The total energy consumption, including support requirements (controls, etc) and recording for 600 seconds amounts to 142 wh, leaving a contingency of 78 wh (total supply 220 wh).

#### 7.10.7.2 Experiment Definition.

Low-g Facility	Rocket Class 1, Traj. A
Number of Expts./Flight	6
Method of Heating	Direct Resistance
Method of Cooling	Passive/Controlled
Apparatus	6 Processing Modules
Apparatus Weight	58 kg
Total Payload Weight	130 kg

#### 7.10.8 Apparatus and Payload Definition

The payload consists of the support module, an atmosphere control system and 6 processing modules. It is identical to the payload defined in 7.8.8 (Superconductors), except for an additional battery pack (4) in the support module. This section is, therefore, limited to a summary of the significant payload data.

##### 7.10.8.1 Processing Modules

Dimensions (cm)	16 x 18 x 14 high
Weight (kg)	12
Max. Power Rating (W)	800

##### 7.10.8.2 Support Module. (Numbers in parenthesis identify components specified in 5.2.2 and Table 5-1)

Basic Structure	33 kg
2 Batteries	6 kg
Power conditioning (5A, 5B)	6 kg
Sequencer, Recorder	4 kg
Contingency	<u>2 kg</u>
Total Weight	51 kg
Net space for apparatus	90 cm (axial weight)



7.10.8.3 Payload Assembly. The payload assembly is illustrated in Fig. 7.10-4

Major data are:

<u>Weights and Space</u>	<u>Weight (kg)</u>	<u>Height (cm)</u>
Support Module	51	60
6 Processing Modules	72	84
Argon Supply System	7	-
	<hr/>	<hr/>
Total Payload	130	144
R-1 Capacity	130	150
Contingency	0	6
<u>Power</u>	<u>Payload Capacity</u>	<u>Required</u>
Total Energy	220 wh	142 wh
Max. Discharge Rate	3000 w	2400 w

In view of the substantial power surplus (78 wh), all check-out tests can be performed with board-battery power.

#### 7.10.9 Experiment Performance

7.10.9.1 Ground Operations. After check-out of all systems, experiment performance starts 2 minutes before launch. Ground operations consist of:

- (1) Activation of board recorder
- (2) Pressurization of processing chambers
- (3) Activation of g-switch.

7.10.9.2 Flight Operations Consist of: (Recorder and chamber pressurization continue uninterrupted to the end of flight operations at 600 sec.)

- (1) Activation of timer by g-switch
- (2) Processing program, as detailed in Sect. 7.10.7.1 (+90 to +330 sec)
- (3) Passive terminal cooling
- (4) Deactivation of payload at +600 sec.

7.10.9.3 Post-Flight Operations. Consist of the recovery of the samples and flight recordings and experiment evaluation.

7.10.9.4 Time Diagram. The sequence of operations is detailed in the time diagram, Fig. 7.10-5.

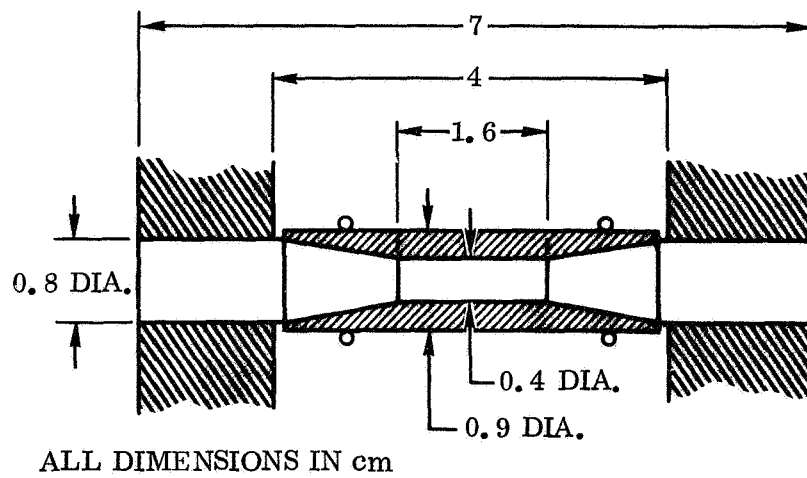


Figure 7.10-1. Sample with Ceramic Sleeve for Metastable Alloy Experiments at High Temperatures

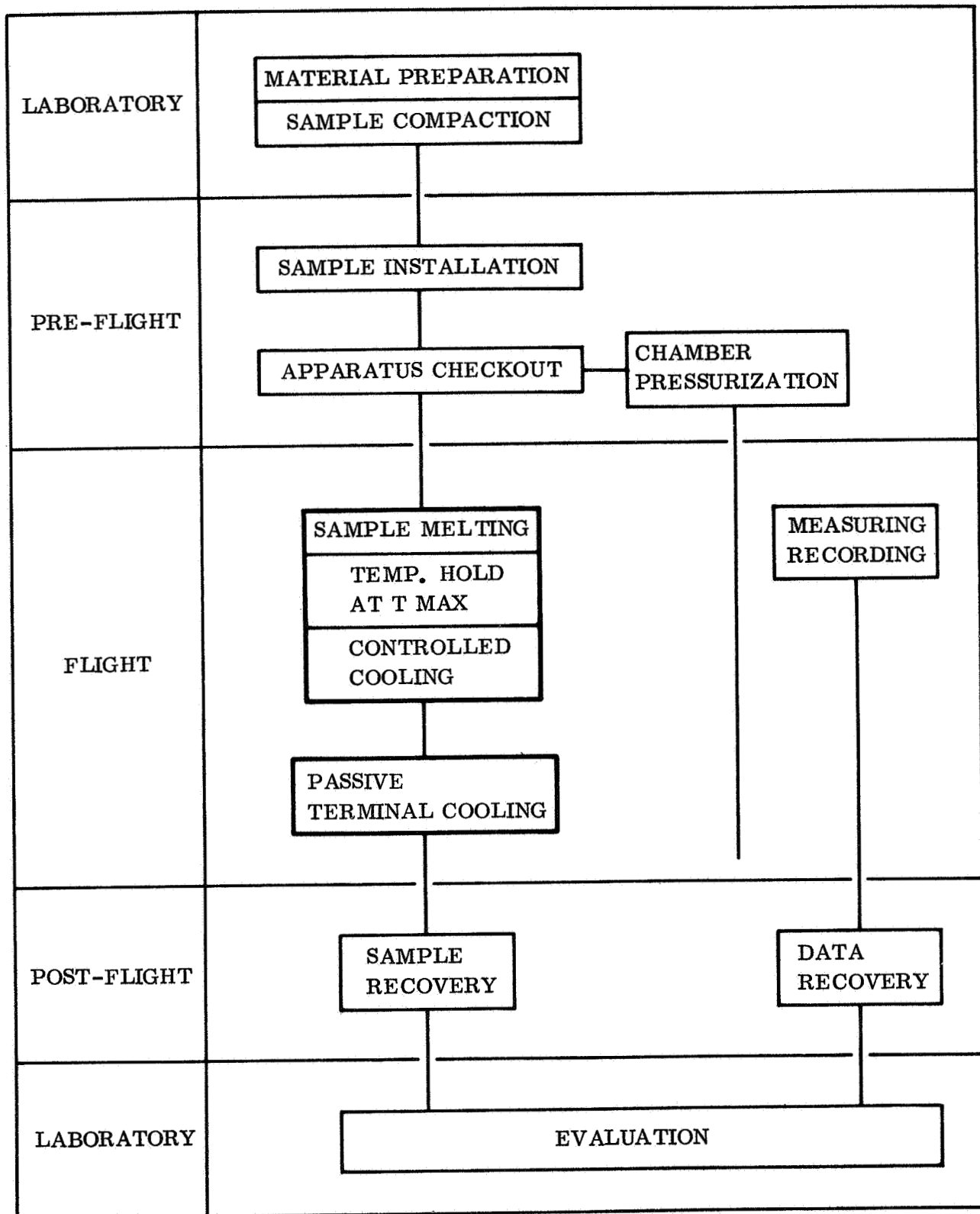


Figure 7.10-2. Process Flow Diagram - Metastable Alloys -  
High Temperature Thermal Dispersion

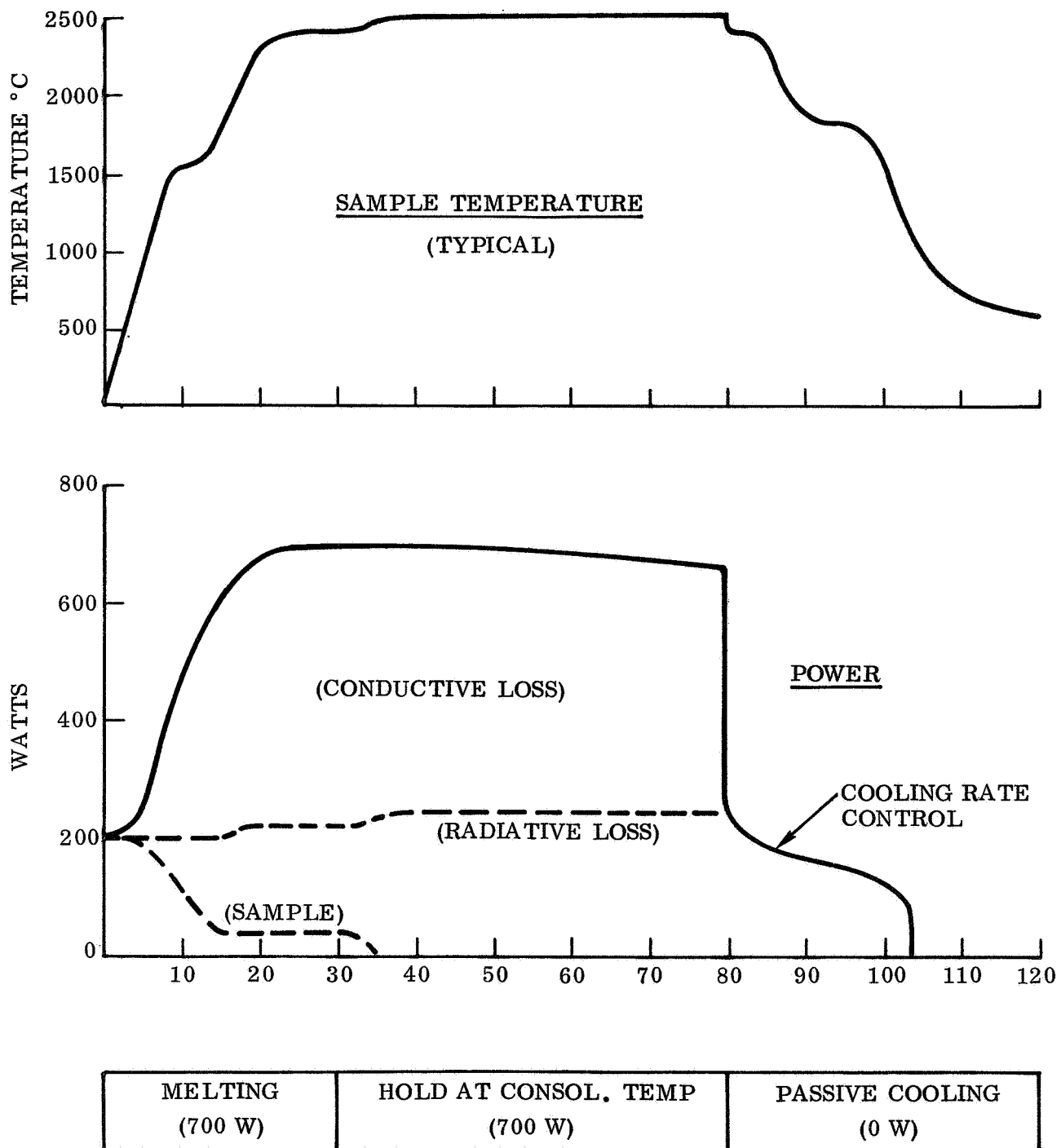


Figure 7.10- 3. Heating Profile for Metastable Alloys

## ROCKET PAYLOAD ASSEMBLY

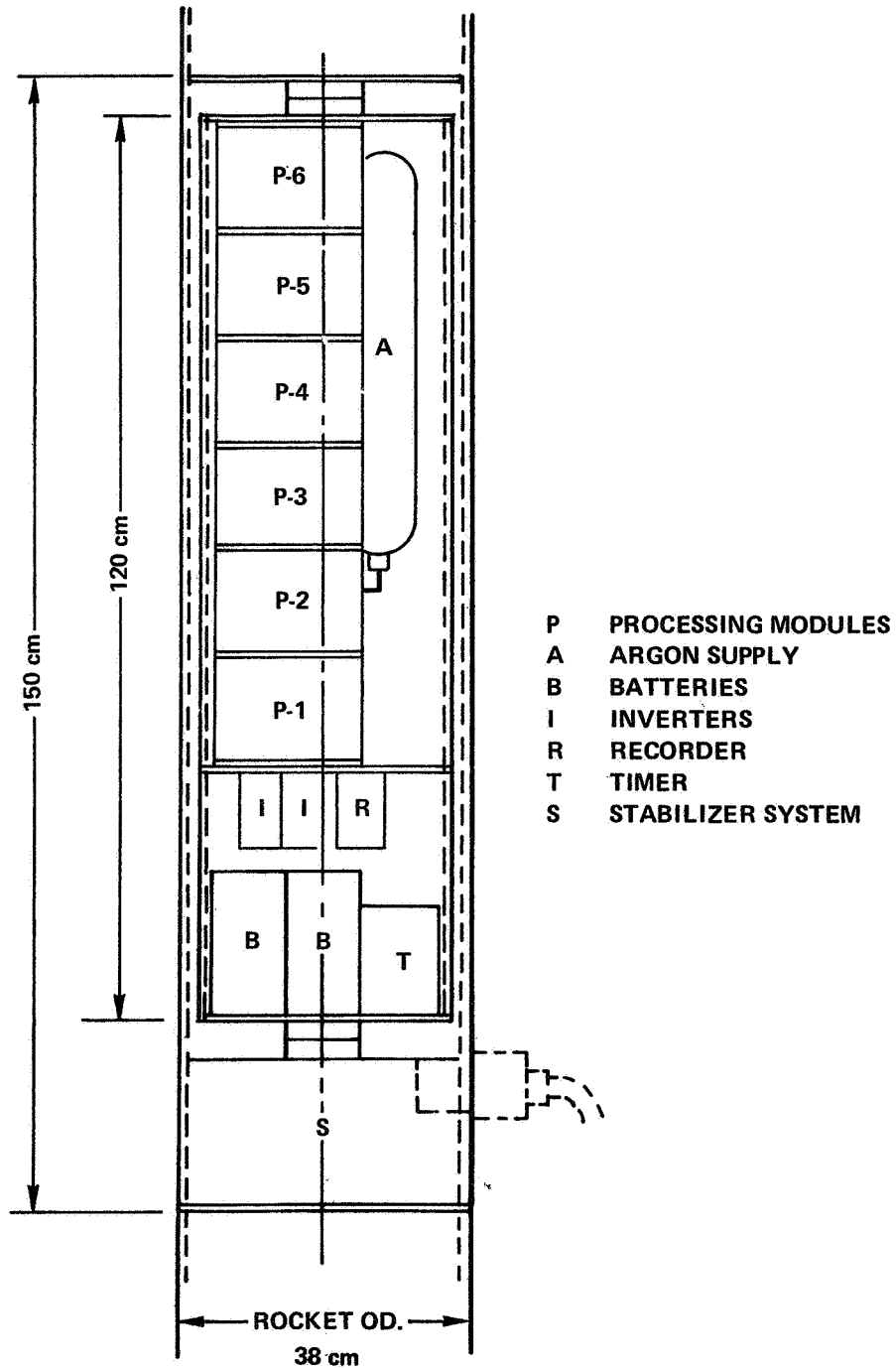


Figure 7.10-4. Payload Assembly — Metastable Alloys (Thermal Dispersion — High Temperatures)

# EXPERIMENT PROGRAM – TRAJECTORY A

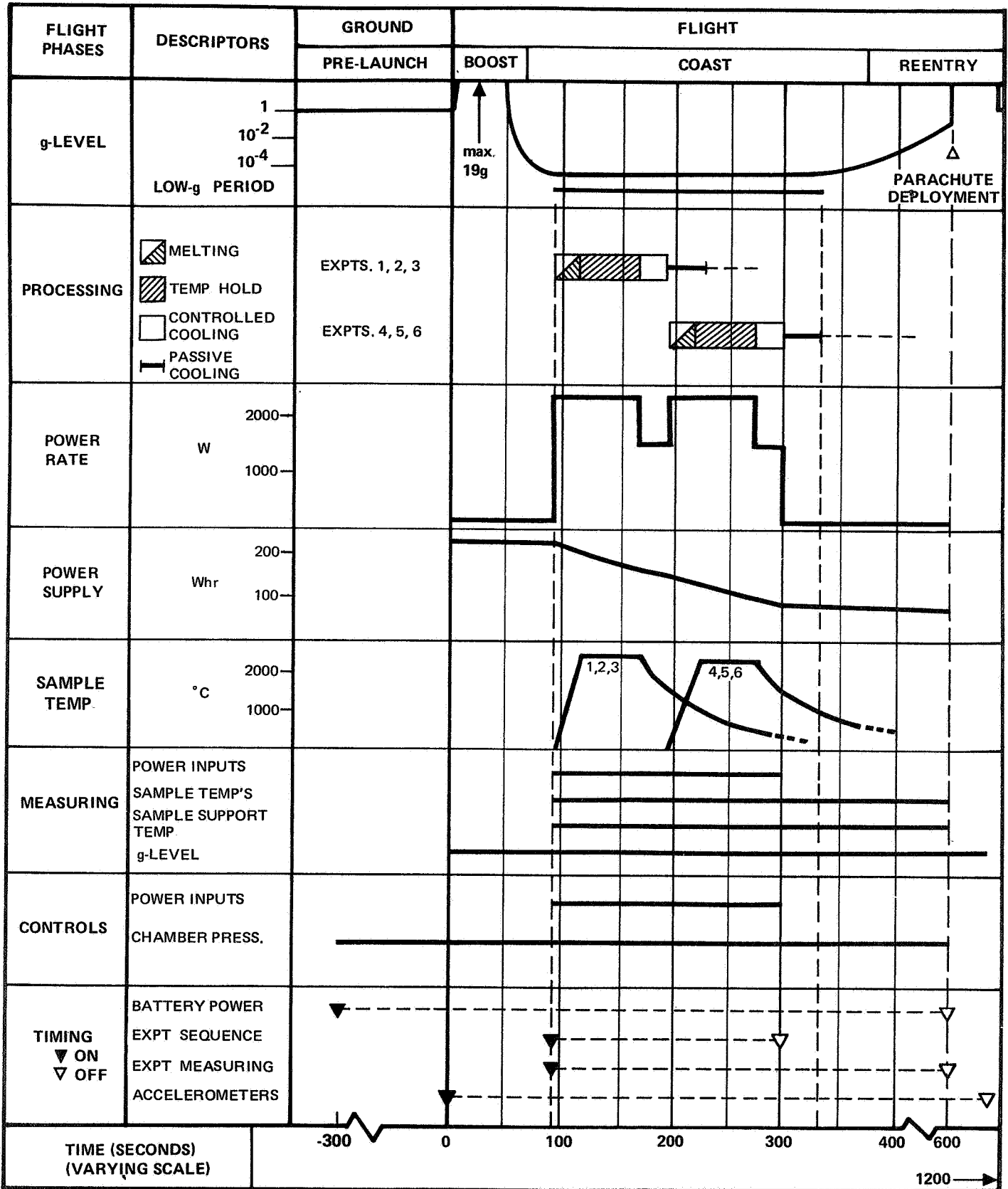


Figure 7.10-5. Metastable Alloys – High Temperature Thermal Dispersion

## 7.11 METASTABLE ALLOYS - HOMOGENIZATION

In this process it is attempted to enhance the dispersion and the related properties of metastable alloys by mechanical agitation during the cooling period to solidification. As pointed out in the introductory discussion of metastable alloys (Sect. 7.9.1), experiments are limited to the moderate temperature regime (1000° C max. ). Aside from the extensive leadtimes, the development of high temperature homogenization techniques should await the results of the proposed experiments at lower temperatures.

In principle, the process is identical to thermal dispersion at moderate temperatures (7.9), except for agitation during the g-sensitive cooling period and for the addition of an agitation device to the processing module defined in 7.9. Therefore, only those subjects are discussed which call for additional definitions or revised data.

7.11.1 - 7.11.3 Objectives, Verification Requirements and Materials as in 7.9, except for the deletion of verification level I; agitation obviously calls for extended liquid-state processing time (Level II).

### 7.11.4 Material Quantity and Sample Size

For homogenization, a cylindrical sample configuration is chosen, since it is more adaptable to the transfer of acoustic energy and to transducer configurations. The cylindrical sample container is divided into two sections, so that two compositions of the same alloy system can be processed in one experiment. The energy transfer through the dividing wall appears to present no problem. The related data are as follows:

#### Sample Container

Diameter	0.8 cm
Total Length	6.0 cm
Length, each section	3.0 cm

#### Material Quantities (Each Half-Sample)

Volumè	1.5 cm <sup>3</sup>
Weight Range	5.8 - 16.0 gr
Evaluation Sample Size	0.8 x 3 cm



### Material Quantity per Experiment

Weight 11.6 - 32 gr

#### 7.11.5 Processing Phases

Identical to 7.9.5, except for added agitation phase parallel and concurrent with the g-sensitive passive cooling phase.

#### 7.11.6/7 Low-g Test Requirements, Facilities and Experiments

As defined in 7.9.6/7, except for a reduction of the number of experiments/flight from 4 to 3, based on the following assessment.

##### 7.11.7.1 Number of Experiments/Flight

###### Payload Weight

4-Expt. Payload	123 kg
Margin (RR-1, RR-4)	2-7 kg

###### Payload Space

Net Space in Support Module	80 cm
4-Expt. Payload	<u>94 cm - unfeasible</u>
3-Expt. Payload	76 cm
Margin/3 Expt. Payload	4 cm

Since space limitations permit a max of 3 expts. per flight, all further data apply to a 3-expt. payload.

7.11.7.2 Power/Time Requirements. The time scheduled for the 3-experiment payloads IIa and IIb are as defined in Section 7.9.7.3, except for battery-supplied power requirements which are as follows:

<u>Process Phase</u>	<u>Energy Rate</u>	<u>Energy Consumption (wh)</u>	
	<u>(w)</u>	<u>IIa</u>	<u>IIb</u>
Ground Heating	(1150-2450)	-	-
Flight Hold at 1000° C	2100	53	53
Pass Cooling/Homogenization	450	23	42

<u>Process Phase (Cont'd)</u>	<u>Energy Rate</u> (W)	<u>Energy Consumption (wh)</u>	
		<u>IIa</u>	<u>IIb</u>
Active Cooling	450	8	8
Terminal Cooling	450	80	80
Total Energy Consumption		164	183
Battery Supply		220	220
Margin		56	37

#### 7.11.7.3 Experiment Definition

Low-g Facility	RR-1, Traj. A or RR-4, Traj. B
Number of Expts/Flight	3
Number of Samples/Flight	6
Method of Heating	Radiation/Electrical Heating Element
Processing	Passive Cooling/Homogenization
Solidification	Circulating Water Cooling

#### 7.11.8 Apparatus and Payload Definition

The payload consists of the support Module and the apparatus for three (3) experiments.

7.11.8.1 The Apparatus consists of 3 processing modules, a central argon supply system and an ultrasonic energy generator located in the support equipment section of the payload can. The processing modules are identical to those described in Sect. 7.9.8.2, except for the addition of the homogenization device. The ultrasonic transducer, measuring 5 x 5 x 4 cm is attached at the top of each processing chamber. The acoustic energy is transferred to the sample with a "horn" reaching into the processing chamber and serving as sample support. The major data for the individual module and the apparatus assembly are:

	<u>1 Module</u>	<u>3-Module Apparatus</u>
Diameter	28 cm	28 cm
Axial Height	23 cm	
" with Gas System		77 cm
Chamber Volume	200 cm <sup>3</sup>	
Max. Power Rating	1000w	2300w
Weight (with coolant)	15.5 kg	54 kg

7.11.8.2 The Support Module is as defined in Sect. 7.9.8.3.

Support Module Weight	57 kg
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7.11.8.3 Payload Assembly. The payload assembly is illustrated in Fig. 7.11-1. Its major data are:

Payload Weight

Apparatus	54 kg
Support Module	57 kg
Total Payload	113 kg
RR-4/B Capacity	125 kg
Margin	12 kg

Payload Dimensions

Max Diameter	38 cm
Max Height	150 cm
Net Space for Apparatus	80 cm
Apparatus Height	77 cm
Margin	3 cm

Payload Power

Max Ground Power Rate	2,450 w
Max Flight Power Rate	2,100 w
Stored Board Power	220 wh
Max. Flight Consumption	183 wh
Contingency	37 wh

#### 7.11.9 Experiment Performance.

Identical to 7.9.9, except for the addition of homogenization concurrent with passive cooling (7.9.9.2, step 3).

# ROCKET PAYLOAD ASSEMBLY

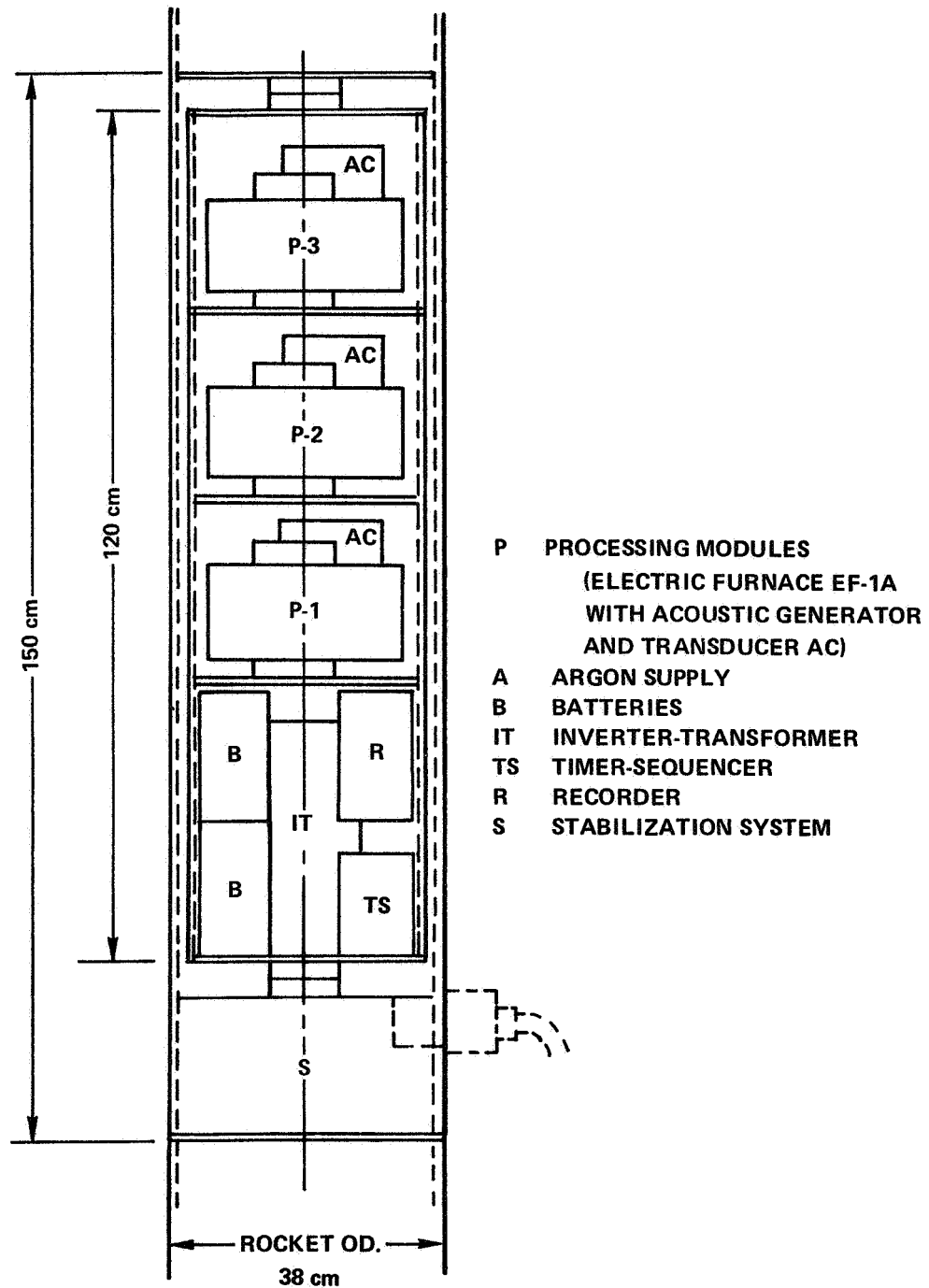


Figure 7.11-1. Payload Assembly — Metastable Alloys — Homogenization

## 7.12 SINGLE CRYSTAL GROWTH

### 7.12.1 Process Definition and Objectives

The development of solid state technology has led to a demand for single crystals of ever greater size, purity and perfection. This demand is especially urgent in the semiconductor field. Tons of silicon and germanium must be processed and grown into single crystals for the manufacture of transistors, diodes, rectifiers, meters, thermoelectric devices, magnetic switches, detectors, filters and numerous other devices.

The preparation of large single crystals may be said to consist of two basic processes:

- (a) Purification of material
- (b) Growth of the crystal

Impurities in concentrations of less than one part per million have pronounced effects on the semiconducting properties. Purification techniques involve processes such as zone melting, distillation, sublimation, filtration, electrolysis, extraction, etc. The material resulting from the purification treatment is then subjected to growth into a single crystal.

The crystals are commonly grown by seeding melts of the desired composition. Sometimes the purification and the crystal growth are performed simultaneously by using zone melting techniques. These consist of causing a melted zone to traverse a length of sample by producing relative motion between the sample and a localized heat zone. Purification occurs if foreign material is more soluble in the liquid phase than in the solid. The single crystal is attained by appropriately positioning a seed with its orientation directed for growth along the desired axis. Growth rate is controlled by the temperature gradient at the hot zone and the relative rate of motion between hot zone and the crystallizing material.

Because most materials undergo a volume change when they change state from liquid to solid, buoyancy and thermal forces tend to disturb the natural growth of crystals in normal gravity. Gravity may cause small growing crystals to break off and induce

multiple nucleation. Thermal gradients may cause the generation of convective currents which will distort the growth pattern and cause imperfections in the crystal. In zero-g these effects are unimportant or inoperative so that the larger more perfect crystals may be expected. It is also possible to manipulate material without wall contact in zero-g, so that contamination from the container is eliminated as well as thermal stresses from differential expansion and contraction between the material and the container wall. Thus, stress-originated cracks, dislocations, strains, lattice vacancies, and other imperfections are avoided. Other perturbations include grain boundaries, segregates and inclusions.

The objective of this experiment therefore is to prepare large pure single crystals with enhanced properties for application in piezoelectric, ferroelectric, dielectric magnetic, electrooptic and acoustic devices. In initial experiments single crystals will be prepared in low-g and the expected improvements in growth perfection will be evaluated by comparing the results with identical samples prepared in one-g. The potential for increasing the rate of growth will also be examined.

#### 7.12.2 Verification Requirements

The advantage of growing crystals in zero-g will be evaluated by measuring and comparing the properties of crystals grown on earth with the same crystal material grown in space. Success will be measured by the crystal quality and crystal purity, with controlled addition of selected dopants for some materials. The evaluation entails procedures ranging from simple visual inspection to x-ray diffraction and beam width measurements.

Visual inspection alone is sufficient to detect gross defects. Among these are voids, cracks, twins, haze (in transparent crystals), crazing, spikes, polycrystallinity etc. This examination is done under oblique light, and is quantified by subsequent counting of the defects under a microscope.

Electrical and optical measurements are used to determine the purity of the material. The resistivity and the Hall effect measurements indicate the number of impurities and give some indication of their type. Carrier mobility and concentration may be calculated from the electrical measurements.

Dislocation levels are usually obtained by etching and counting the etch pit density.

The detailed atomic structure is the ultimate criterion of crystal perfection.

Detailed structure will be shown by examining the x-ray diffraction pattern of selected discs cut from the crystal. The x-ray beam width, or scatter, also gives a measure of the number of imperfections.

Many semiconductor materials are optically active as filter and detector devices. Simple infrared absorption measurements quickly give indications of purity and fault concentrations.

The material properties of importance in the evaluation may be summarized as follows:

- (a) Visual inspection for gross defects
- (b) Electrical conductivity, and its temperature effect
- (c) Hall coefficient, and its temperature dependence
- (d) Carrier mobility
- (e) Carrier concentration
- (f) Etch pattern
- (g) Optical transmission
- (h) X-ray diffraction pattern and beam width
- (i) Elastic modulus
- (j) Tensile strength

The last two properties above are of importance for single crystal whiskers and perhaps bulk ingots which will eventually be produced.

Measurement and correlation of the above properties for zero-g and one-g grown crystals will determine the advantages of low-g preparations. These advantages must then be generalized inasmuch as the ultimate goal is a product which will improve our knowledge and lead to better and more effective devices.

The best approach for reaching the goal of improved single crystals is to proceed stepwise in complexity and difficulty of experiments. Initially, material easy to obtain as a single crystal will be investigated. The melting temperature should be relatively low so that heating and containment problems are minimal. A low melting, easily



produced semiconductor is therefore a good candidate for preliminary verification of low-g processing.

The results, conclusions and predictions obtainable from low-g processing will be more valid and of wider utility by comparing results of experiments which manifest a range of potential thermal and density gradients. Therefore results with crystal having easily attained low melting points will be compared with results from experiments with higher melting crystals. Steeper gradients prevail during the processing of these higher melting crystals.

The high temperature material must resist reaction with container materials and with normally used atmospheres. Operation at elevated temperatures will also provide a test for preventing oxidation and for testing effectiveness of handling procedures.

A still higher level of verification entails the manufacture of very large single crystals. It requires more time than is available during a drop tower test or rocket flight-namely a Skylab station. This is necessary because large masses of perfectly formed crystals demand slow, carefully temperature-regulated time for growth, which is not available in suborbital flight.

Two immediate levels of verification can therefore be identified plus one adapted to orbital flight:

#### Verification Level I

Verification of single crystal growth and perfection by remelt and recrystallization of a low temperature easily obtained semiconductor. The electrical, chemical and optical properties will be correlated with similar material processed in one-g.

#### Verification Level II

Determination of the difference in properties between a semiconductor with an elevated melting point when processed in low-g and in one-g. Again, the criteria will be the improvement in electrical, chemical (etching) and optical properties.

#### Verification Level III

The ultimate verification level will be to process a large single crystal in space using

commercial quantities of material in order to confirm that the expected benefits are obtained and space production is feasible.

### 7.12.3 Experiment Materials.

The criteria for selecting the experimental materials is as follows:

- (1) Must be a useful semiconductor.
- (2) Properties must be well characterized.
- (3) Possess a low or easily obtained melting point.
- (4) Crystal form should be readily achieved.

There are many candidate materials which may be considered. The technology for their preparation is now rather well prescribed. Many are made on a production basis. This is also the goal of low-g processing -- commercial production. A well known single crystal compound which has a conveniently low melting point is indium antimonide, InSb. It is easy to prepare and has a melting point of  $530^{\circ}$ . Applications include filters, detectors, transistors etc.

Success and/or information gained from the InSb single crystal experiment will provide the foundation for extending the preparations to higher temperature materials of greater size. For this purpose, lead sulfide, PbS melting at  $1114^{\circ}\text{C}$  is selected. This semiconductor is a widely used detector. No problems because of vaporization of one of the components will occur nor will toxic materials be involved. PbS is well characterized and provides an excellent basis for evaluation of the success of the experiment.

A wide choice of other candidates is available. Selections may be made according to application (ferroelectric, piezoelectric, electrooptic, etc.), chemical type (elements, compounds) or melting temperature. The following materials are offered, not as specific candidates at present but as typical examples of material types which are suitable for experiments at low-g:

<u>Name</u>	<u>Type</u>	<u>M. Pt.</u>	<u>Application</u>
Si	Element	1420° C	Semiconductor devices
Ge	Element	940° C	Semiconductor devices
GaAs	III-V Compound	1280° C	Sight emitting diode, detector
ZnTe	IV-VI Compound	1240° C	Electro optics
Mg <sub>2</sub> Sn	II-IV Compound	778 ° C	
BaTiO <sub>3</sub>	Ternary Compound	1600° C	Piezoelectric

#### 7.12.4 Material Quantities, Sample Size and Heating Method

The objective of this experiment is to produce enough material to show that desirable new and enhanced properties are obtained from crystals grown in low-g.

7.12.4.1 Target Material Quantity . Enough material must be obtained to enable tests to be made which verify the properties of the product obtained. The critical tests such as the electrical conductivity and Hall coefficient may be made with very small quantities of material consisting of thin discs with an area of a few square millimeters. The gross inspection of the solid product requires the largest possible single crystal, of course. Moderately small samples are tolerable for elastic modulus and tensile tests.

The greatest restrictions on the sample size and configuration are imposed by the temperature and the heating methods for a test period which is relatively short. The size and shape selected to meet the requirements of providing significant data on the zero-g effects and still be readily processed by heating and cooling is one consisting of a cylinder of material. The length and diameter are determined by the temperature of processing, the material conductivity and the heat transfer environment. Accordingly the following sizes are used:

<u>Verification Level</u>	<u>I (InSb )</u>	<u>II (Pbs )</u>
Shape	Cylinder	Cylinder
Length	8.0 cm	8.0 cm
Diameter	0.4 cm	0.4 cm
Volume (cm <sup>3</sup> )	1.005	1.005
Weight (gr)	7.33	7.54
Heat content	32 cal (30° - 550° C)*	63 cal (30° -114° C)*

\*For 0.8 cm heated section only

7.12.4.2 Heating Method. The heating method selected for melting the "preform" of the single crystal must provide a uniform temperature environment surrounding the sample. Any local nonuniformity will be reflected in uneven heating and cooling of the sample resulting in undesirable nucleation at cool sites. The desired crystallization process is that which arises at the solid, unmelted, properly orientated cool end portion of the sample cylinder which acts as a seed.

Radiant heat must produce the melting. Convective heating which can introduce impurities is neither desirable nor appropriate, and conductive heating is not feasible for semiconductors because of the following:

- (a) Their resistance is too high
- (b) Passing current, especially the high heating currents necessary for melting disrupts the structure of the solid.

The melting is customarily accomplished by inductive heating or by resistance heating.

The complexity and weight required for inductive heating is too great for the present application. Resistance heating has been selected therefore and may be accomplished by:

1. Wire heating elements embedded in ceramic
2. Exposed wire heating elements in a ceramic base
3. Cylindrical metallic radiator

The best method for heating semiconductor rods is the use of the latter method, a heated cylindrical metallic tungsten element. This method shown in Fig. 7.12-1 has the following advantages:

- (a) It can be made rapidly responsive
- (b) It can be made uniform
- (c) It is non-contaminating
- (d) It can be made light weight using AC power

After melting is attained, the cooling cycle begins. It must be controlled so that solidification proceeds in one direction through the melt, beginning at the cooler end which acts as the seed. To accomplish this, the heater is attached by two brackets of unequal size and heat capacity. These also serve as the heater electrodes. The more massive electrode acts as a heat sink so that recrystallization proceeds from this end towards the opposite end which stays warmer.

The sample is firmly fixed and immovable at one end. The opposite end is also set into a firm bearing surface but is not fixed firmly. It is therefore free to move in response to forces which tend to expand the sample.

7.12.4.3 Sample Configuration and Size. The basic sample configuration is shown in Fig. 7.12-1. The sample is heated by radiation from a cylindrical tungsten heating element surrounding the sample. The heating zone of the element is achieved by thinning the tungsten cylinder to produce a section of high resistance. The length of the heating section is determined by the criterion that the surface tension of the molten semiconductor will prevent its separation from the fixed solid portion of the rod of diameter  $d$  provided the length of the liquified portion does not exceed  $\pi d$ . A conservative calculation leads to a configuration which gives a heated zone of 8 mm for a sample diameter of 4 mm.

The same sample configuration is used for verification level II. The sample for this experiment has a higher melting temperature and therefore requires a heater with greater output, but the configuration remains the same. The required configuration and weight is therefore the same as given in Section 7.12.4.1.

#### 7.12.5 Experiment Process Phases

This is essentially a very simple experiment to perform because it amounts to a re-crystallization of a preformed solid material.

7.12.5.1 Pre-test (ground) Operations. The ground operations consist of the following steps during which cleanliness and attention to avoid contamination are of utmost importance:

1. Select the raw sample
2. Orient the crystal to achieve desired seeding, then cut to size
3. Mount sample in heater, fixing ends firmly
4. Assemble apparatus, install, and check-out

The number of transfer and handling operations should be kept to a minimum. The manipulations are best carried out in a clean-box with instruments used solely for one material.

#### 7.12.5.2 Test (low-g) Operations

The low-g operations consist of

1. Heating and melting the sample
2. Recrystallize by radiation cooling

The heating and cooling are done in an atmosphere of purified argon to avoid introducing contaminants. No manipulations are required but it is important to allow no vibrations or accelerations which can disturb the liquid zone and cause spurious nucleation, or distorted and defective crystals. The seed or nucleus for the recrystallized material exists at one end of the solid position of the unmelted sample. The orientation and cutting of the installed sample is done with care to assure that crystal growth proceeds in the desired direction.

7.12.5.3 Post-Test Operations . The processed sample with a central recrystallized portion is removed and evaluated for zero-g effects using the tests identified in Section 7.12.2. These consist chiefly of visual inspection, measurement of Hall

coefficient, resistivity, x-ray pattern and calculation of carrier mobility and concentration.

7.12.5.4 Flow Diagram. The processing sequence is identified in the flow diagram Fig. 7.12-2 and the preliminary time diagram, Fig. 7.12-3.

#### 7.12.6 Low-g Test Requirements

7.12.6.1 Low-g Time Requirements. The low-g time requirement for the formation of a single crystal precludes the use of a drop tower. Although molten small crystals may be made in a short time, the scaling effects and testing of any product is not conducive to good results. It is feasible to produce a single small almost perfect crystal and yet not be possible to produce a larger one. Modest quantities of material require longer low-g periods for crystal growth. Slow growth favors the desirable large crystals because purification proceeds by the diffusion of impurities ahead of the solidification front. Diffusion is a slow process. Fast cooling creates strains and imperfections like vacancies and dislocations in the growing crystal. Solidification must therefore be reasonably slow.

A compromise must be made between the size of the crystal and the heating and cooling period available for performing the experiment. The desirable very slow crystallization times must be accommodated in a scientific passenger pod or in an orbital vehicle.

For initial experiments in a rocket, a maximum period out of about 390 seconds of low-g time provided by an Aerobee can be used. No power interface on the ground is necessary if the heating is started during 90 seconds of the acceleration period. Additional power for period of 60 seconds can then be used to melt the crystal when low-g is attained. This leaves approximately 240 seconds for the solidification.

Because seeding is inherently present, when cooling begins the crystal of InSb can be propagated at the relatively high rate of growth of 2 mm per minute without twinning or similar defects. The low-g processed material would thus be about 5 mm long. For crystals with low dislocation counts, rates of 0.1 mm per minute or less are preferred. This is also the preferred rate for PbS crystallization.

The power requirements necessary to achieve the crystallization times above may be calculated by accounting for the following heat sinks:

1. Heat to raise sample temperature
2. Heat to melt sample
3. Heat lost from surface by radiation
4. Heat lost from ends by conduction.

The heating and melting requirements for the 8 mm long sections which undergo recrystallization were shown to be 32 and 63 calories for InSb and PbS respectively in Section 7.12.4.1. Because of this low thermal requirement, heating during prelaunch is therefore not necessary. Gentle preheat is desirable, however so that initial heating may begin at launch and continue for 90 seconds. The thermal power required for the sample to reach the melting point during this time is 19 watts for InSb and 81 watts for PbS. These values account conservatively for conduction losses and for radiation losses. The power consumed by the heater must be about twice this quantity if it is assumed that the narrow annulus and close coupling between the heater and sample provide ideal thermal exchange between heater and sample and that one side of the heater radiates to ambient temperature. If emissivity is neglected heater power requirements are 40 watts and 200 watts for InSb and PbS respectively.

In order to prevent thermal strain during cooling, heat must be provided to maintain the cooling gradient by offsetting the conduction and radiation losses. If it is assumed that 240 sec of processing time is available then the heating requirements for InSb processing will be about 20 watts and for PbS processing about 100 watts.

The processing profile which accommodates the above mode of operation is shown in Fig. 7.12-6. The profile shows that heating begins at launch. Power is increased after 90 seconds so that melting begins in low g. At the end of melting, power is decreased to provide a gradient of 60 degrees for crystallization. If necessary some heat compensation may be provided. Solidification is at a rate sufficient to allow complete solidification 150 seconds after melting is completed. Gentle cooling with thermal compensation to prevent strains is continued until the gravity level begins to rise at which time heating



power is completely shut off. These data are summarized below:

<u>Verification Sample</u>	<u>I (InSb)</u>	<u>II PbS</u>
<u>Time (seconds)</u>		
Heating	90	90
Melting	60	60
Solidification (low g)	300	300
Terminal Cooling	600	600
Total Low-g Time	390	390
<u>Power/Heat, per experiment sample*</u>		
Max. Input, watts	150	390
Total Energy, wh	22	47
Total Heat, cal.	189000	404200

\*Includes sustaining heat after low-g processing.

The energy requirements for 5 simultaneous samples can be met with one battery (110 wh) verification level I, but 2 batteries are necessary for experiments at verification level II.

7.12.6.2 Required g-Level. The g-level required to achieve significant data must be less than the magnitude of the forces arising from density and thermal gradients. Because volume changes on fusion are of the order of 1 to 50 parts in a 1000, the gravity level should be below  $1 \times 10^{-3}$  at least. The evaluation of the interrelation between thermal gradients and gravity is more difficult to make than this estimate. Experience in the laboratory and during KC-135 tests has shown that levels as high as  $1 \times 10^{-2}$  -g can yield information in some experiments, but this level is undoubtedly high for single crystal tests. Accordingly, the following g-levels may be defined:

Verification level I: A g-level of  $1 \times 10^{-5}$  is the target g-level although a level of  $1 \times 10^{-4}$  g may be acceptable.

Verification level II: A g-level of  $1 \times 10^{-5}$  is desired as a minimum in order to permit assigning observed differences only to the zero-g effects.

#### 7.12.7 Low-g Test Facilities and Experiments

A comparison of the time required to achieve enough crystal growth to provide significant measurements shows that initial information can be obtained from experiments carried out in a research rocket class 4 and the max. standard WSMR Trajectory B. The growth of perfect crystals is a slow process which is not feasible in a drop tower. The materials selected may be grown at the relatively high rate of several millimeters during a rocket flight. This provides measurable sample material. However, more definitive data will be attained when there are longer solidification times available. These advanced tests require a suborbital or Skylab facility.

Two types of single crystal experiments can be performed to test two levels of verification. One level is at a low temperature with an easily obtained crystal. The second level is at elevated temperature at which the density and thermal gradients are enhanced.

It is apparent that the available low-g time of 390 seconds should be fully utilized. Consequently, each sample uses the full time, and experiments are carried out concurrently. The primary criterion for the number of experiments which can be accommodated in one flight is power consumption. The relatively high power requirements are caused by the continuation of heating after solidification (post low-g period) for 600 seconds at a gradually decreasing power rate. A total of 5 experiments per flight in 5 separate processing modules can be carried out, as evidenced by the following evaluation.

##### 7.12.7.1 Power Limitations

<u>Level</u>	<u>I</u>	<u>II</u>
Max Power Rate	150 w	400 w
Consumption		
5 Experiments	110 wh	235 wh
Instrumentation	50 wh	50 wh
Total	160 wh	285 wh
Number of Batteries	2	3

<u>Level</u>	<u>I</u>	<u>II</u>
Power Supply	220 wh	330 wh
Margin	40 wh	45 wh

#### 7.12.7.2 Space Limitations (In terms of axial height), cm

<u>Level</u>	<u>I</u>	<u>II</u>
5 Processing Modules	70	70
Argon Supply System	0	0
Support Module	70	79
Total Height	140	149
Payload Section	150	150
Margin	10	1

#### 7.12.7.3 Weight Limitations, kg

<u>Level</u>	<u>I</u>	<u>II</u>
5 Processing Modules	62.5	62.5
Argon Supply System	6.0	6.0
Support Module	51.0	54.0
Total Weight	119.5	122.5
RR-4/B Capacity	125	125
Contingency	5.5	2.5

#### 7.12.7.4 Experiment Definition

Low-g Facility	RR-4 (Aerobee 200)
Trajectory	B (Max Standard WSMR)
Low-g Time	390 sec
Number of Expts/Flight	5

#### 7.12.8 Apparatus and Payload Definition

The payload consists of the basic support module and the processing modules. The processing module itself is a modified version of the direct resistance module. The changed configuration is in the method of applying heat.

7.12.8.1 Processing Module. Each processing module accommodates one sample. It consists of the following parts Fig. 7.12-4.

1. High frequency transformer
2. Processing chamber
3. Sample assembly

The transformer consists of a single turn secondary winding especially designed to provide high current. It is attached to the processing chamber. This chamber is blanketed with argon maintained at 1 - 1.5 atmosphere by means of a relief valve.

The sample assembly consists of a cylindrical tungsten heater which completes the secondary circuit of the transformer and surrounds the sample. The heater is attached to the secondary u-shaped turn of the transformer by means of copper electrodes of relatively high mass. These act as heat sinks. They are of unequal size to induce preferential cooling and solidification at one end of the crystal.

The heater is shaped and formed so that only a 1 cm midsection has the high resistance for heating. The crystal is fixed at the high-mass end and has a packing of high conductivity metal-ceramic. The opposite end is firmly attached in a bearing also but can be moved in this bearing by thermal expansion forces.

To avoid wide ranging thermal excursions and to provide a leveling-effect on the energy exchange, the heater is surrounded by a layer of insulation of which it comprises the inner wall.

The size and weight of each unit in the system together with the power requirements are:

Dimensions	16 x 18 x 14 cm high
Weight	12.5 kg
Max. Power Rating	400 watts

7.12.8.2 The Apparatus is identical for levels I and II. It consists of 5 processing modules and an atmosphere control system (argon). Weight and space data are as follows:

	<u>Weight (kg)</u>	<u>Height (cm)</u>
5 Processing Modules	62.5	70
Argon Supply System	<u>6</u>	<u>0</u>
Total	68.5	70

7.12.8.3 Support Module. The support module is similar to that used for all the experiments requiring heating and is composed of the units in the following table together with their weights (Numbers in parentheses identify components as specified in Sect. 5.2.2 and Table 5-1).

	<u>Level I</u>	<u>Level II</u>
Basic Structure	33 kg	33 kg
Batteries	6	9
Power Conditioning	6	6
Programmer, Recorder, Misc.	<u>6</u>	<u>6</u>
Total Weight	51 kg	54 kg
Axial Height	70 cm	79 cm
Power Rate, Max.	150 w	150 w
Total Support Consumption	50 wh	50 wh

7.12.8.4 Payload Assembly. The payload assembly for the single crystal experiments is shown in Fig. 7.12-5. Major data are as follows:

	<u>Level I</u>	<u>Level II</u>
Weight (kg)	119.5 kg	122.5 kg
Height	150 cm	150 cm
Max. Power Rate	300 w	550 w
Total Stored Power	220 wh	330 wh

### 7.12.9 Experiment Performance

This experiment requires very few operations because it is essentially a remelt type operation.

7.12.9.1 Ground Operations. The samples will have been configured and loaded into the heater prior to transportation to the launch site. At the launch site the following operations occur:

1. Check assembly and confirm operation
2. Load environmental gas bottle (argon)
3. Check gas, power, and control circuits
4. Load experiments
5. Check all operations

7.12.9.2 Test Operations. Flight operations consist essentially of actuating environmental controls and energizing the heating circuits. These operations are programmed and proceeds as shown in the time diagram Fig. 7.12-6.

7.12.9.3 Post Test (Ground) Operations. These operations are essentially package recovery and removal as follows:

1. Recover experiment package
2. Remove experiments
3. Recover samples under "clean" conditions
4. Retrieve processing data
5. Remove sample to lab for evaluation.

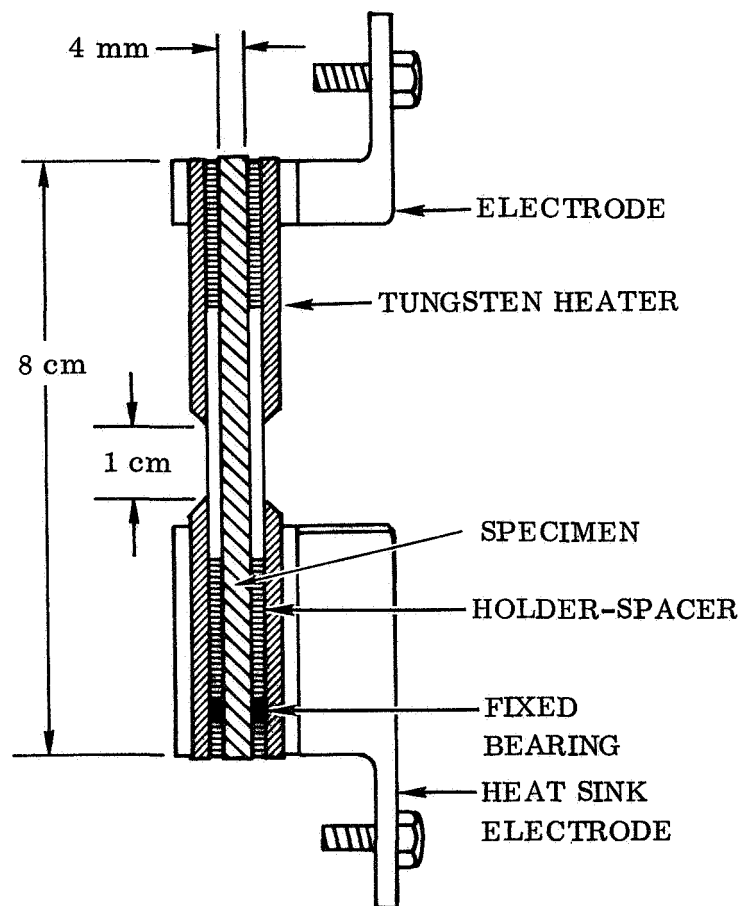


Figure 7.12-1. Sample and Heater

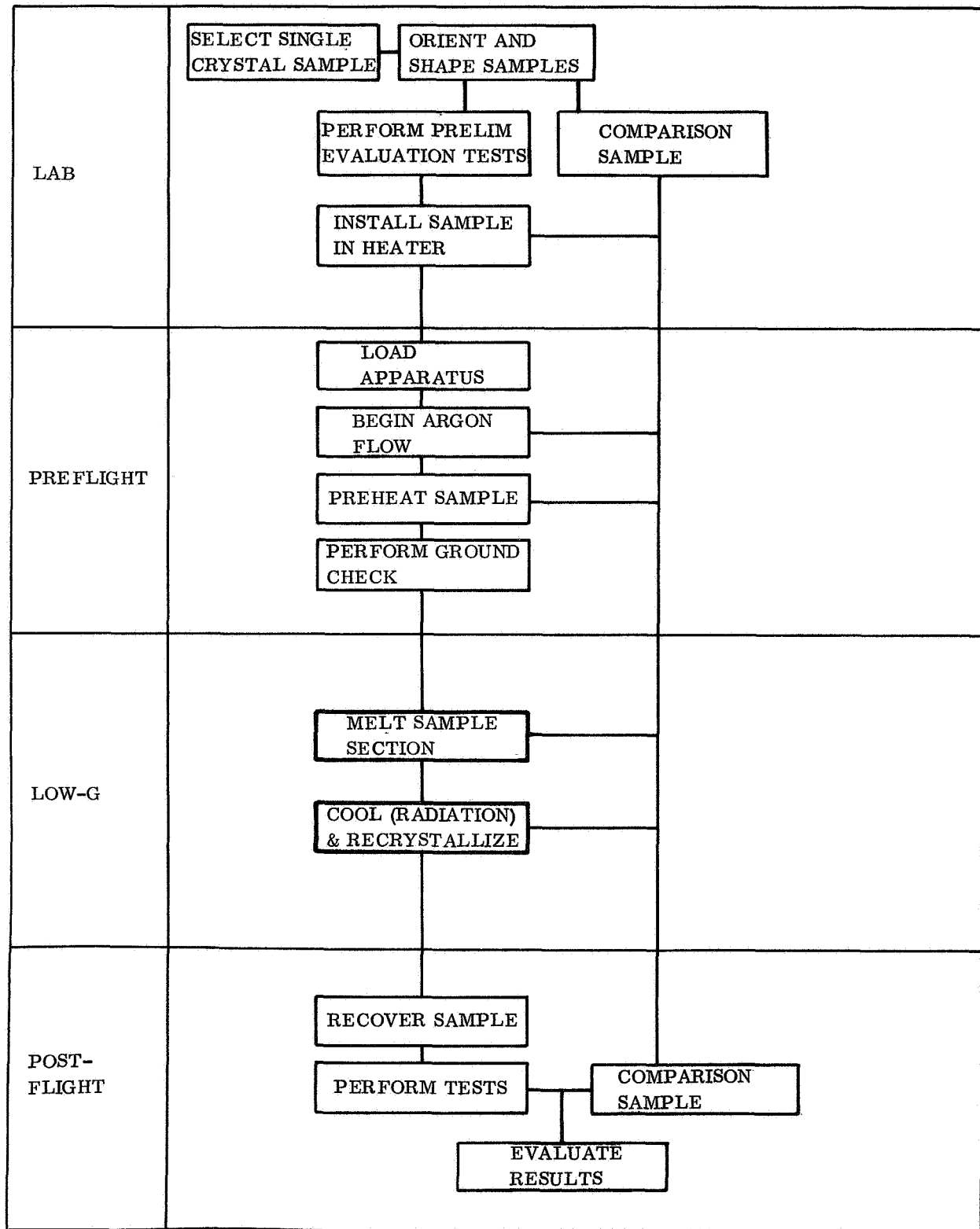


Figure 7.12-2. Flow Diagram for Single Crystal Experiment  
g-Sensitive Phases: Bold Frames



	TOTAL FLIGHT TIME
PRELAUNCH	LOAD SAMPLES AND CHECK APPARATUS
LOW-G TIME	████████████████████
ENERGIZE POWER	Δ
PREHEAT	████████████████
MELT	████████████
COOL	████████████████████
POWER OFF	Δ
POST LAUNCH	RECOVER SAMPLE AND EVALUATE

Figure 7.12-3. Preliminary Time Diagram

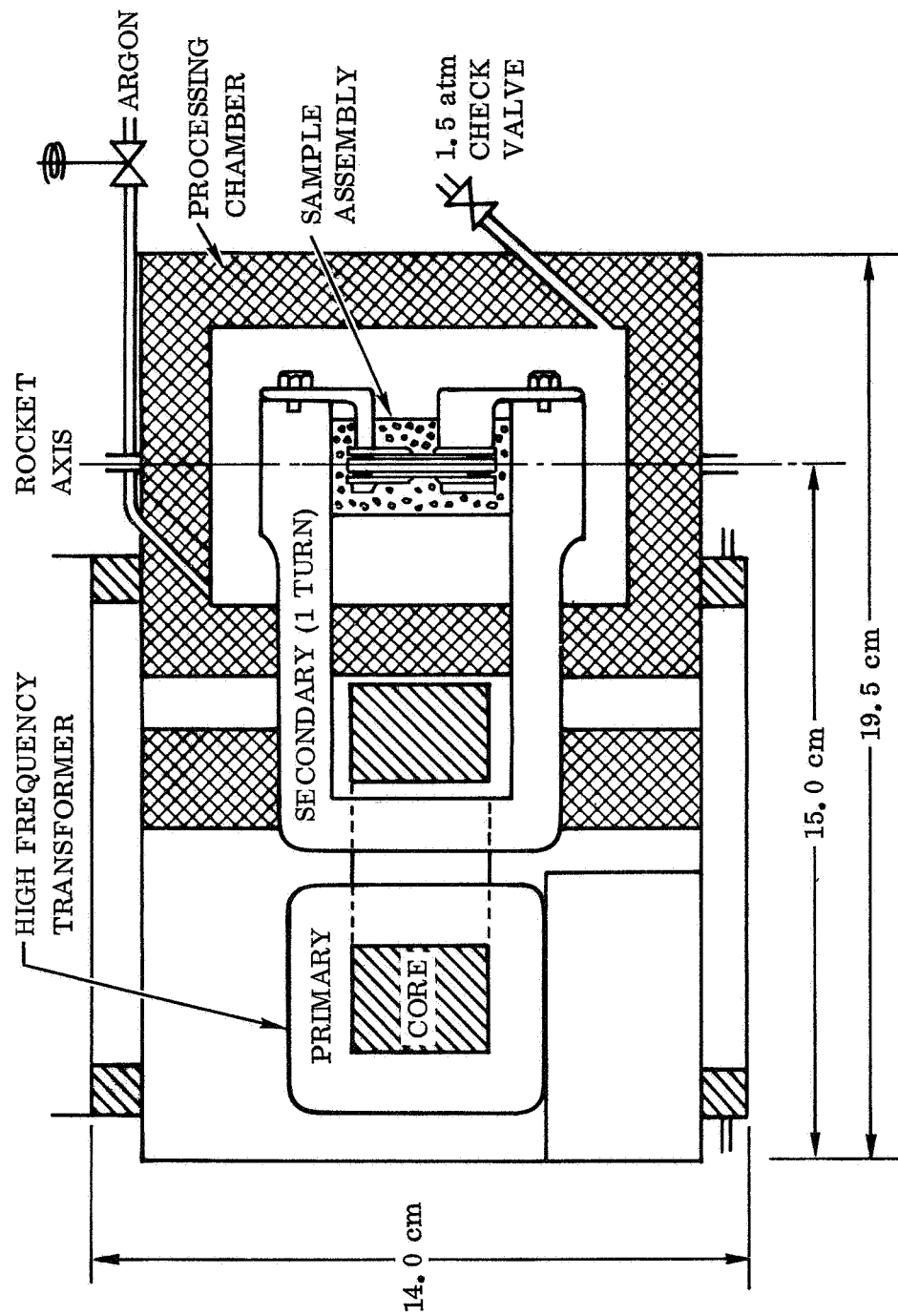


Figure 7.12-4. Radiant Heating Module

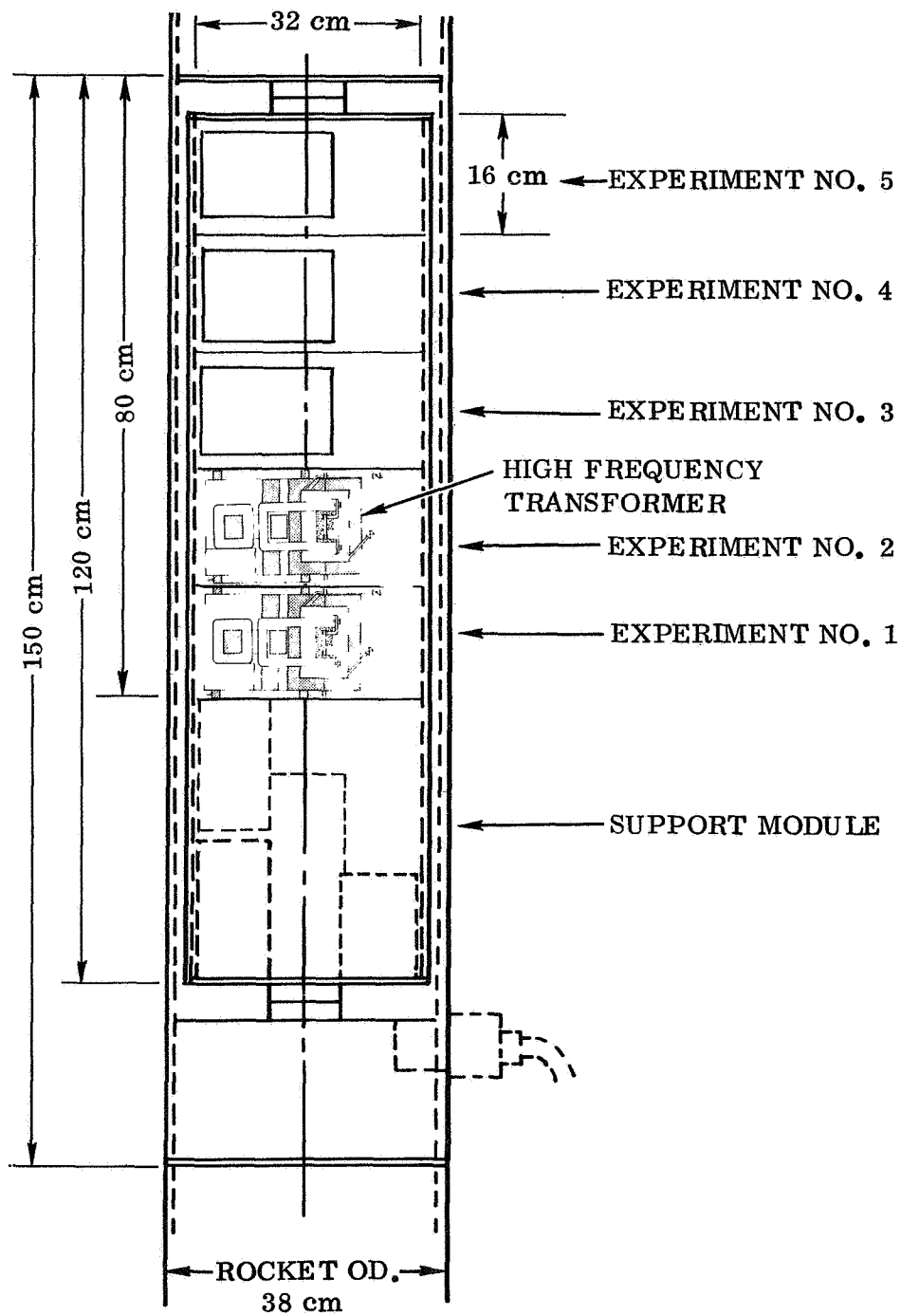


Figure 7.12-5. Single Crystal Experiment Assembly

# EXPERIMENT PROGRAM – TRAJECTORY A

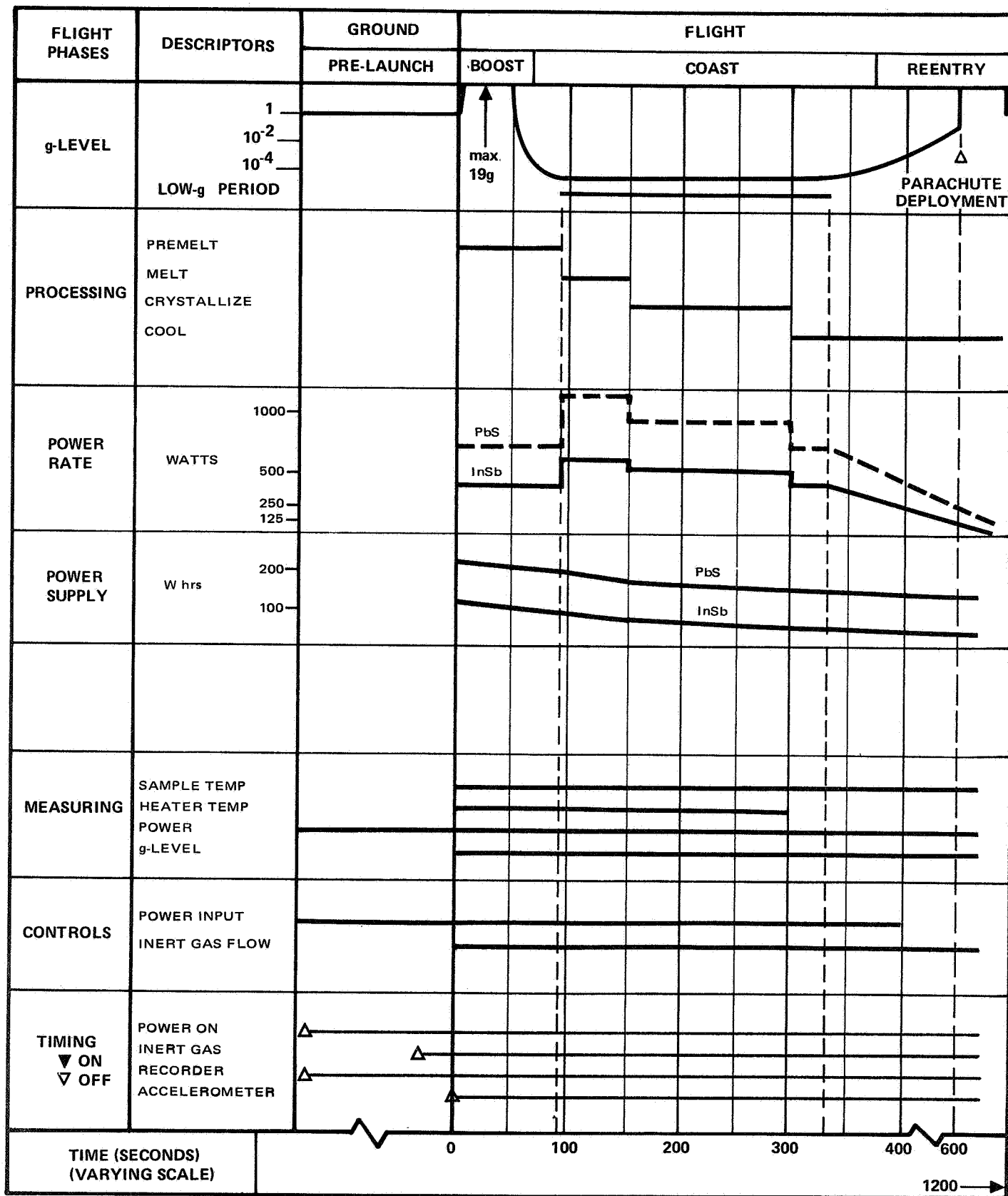


Figure 7.12-6. Time Diagram~Single Crystal Experiment

## 7.13 KINETICS OF NUCLEATION AND CRYSTAL GROWTH

### 7.13.1 Process Definition and Objectives

Nucleation that takes place at random within homogeneous elements of volume of a phase is called homogeneous nucleation. Experience indicates that transformations of nuclei often occur preferentially at various interfaces of the system such as free surfaces, internal surfaces, container walls, suspended crystallites, etc. Such interfaces are said to catalyze nucleation, and this process of nucleation is designated heterogeneous nucleation.

The transformation which occurs when a melt of a pure element, an alloy, or a compound changes to a solid is not a well understood phenomenon in spite of the recent attention given to this process. The production of large, perfect, pure crystals for electronic applications and the improvement in properties arising from controlling the grain size of newer alloys depend greatly on understanding in a microscopic scale the way the atoms or components of a liquid above its melting temperature change to a solid as heat is removed.

The liquid state is characterized by a situation in which the atoms or molecules of which it is composed are relatively free to move, so that over a period of time, the near neighbors are changed many times. In a solid the atoms or molecules rarely change position with respect to neighbors. In order to cause the particles to remain fixed relative to each other, the following free energy changes must occur.

1. Free energy change per unit volume. A liquid volume disappears and a solid volume, not necessarily the same, appears.
2. Interfacial free energy. An interface appears between the liquid and solid.
3. Strain energy. This energy is associated with maintaining the constituents of the solid in a fixed array despite distorting forces.

The above three types of energy control the rate of nucleation. The subsequent growth of the solid nuclei is usually controlled by two types of diffusion:

1. Diffusion of matter. This will limit the growth rate if the solid and the liquid differ in composition.
2. Diffusion of heat. This can be the limiting factor if there is no composition change during the phase change.

Homogeneous nucleation is difficult to achieve. Water, if pure, can supercool to  $-40^{\circ}\text{C}$  without solidifying. Pure metals can supercool a few hundred degrees without nucleating, and alloys may supercool tens of degrees. The difficulty is caused chiefly by the new surface or interface being formed. Eventually, nucleation occurs by spontaneous formation of crystallites because of fluctuations in composition or in degree of aggregation of elementary constituents into "embryos" which may then grow (or dissolve).

Progress in the study of homogeneous nucleation of supercooled liquids was slowed for a long time by two principal experimental difficulties:

1. It is difficult to prepare liquids that are free of minute solid impurities that may serve as foreign nuclei or "seeds" to catalyze the nucleation at small supercooling.
2. Once the bulk supercooled liquid had been seeded, solidification by growth of the nucleus ensued so rapidly that the experiment ended as soon as the first nucleus formed.

Vonnegut solved these problems by reasoning that if the bulk liquid were divided into small droplets which were isolated from each other, the active catalysts would be sequestered in a few of the droplets and the effect thus restricted to a small fraction of the total mass. Using this technique he was able to obtain nucleation kinetics.

Homogeneous nucleation has long been recognized by analytical chemists as the method for obtaining larger, more perfect crystalline precipitates (so that impurities are not occluded). Proper technique is to control nucleation by "adding a dilute solution of the precipitating agent slowly with stirring." This avoids local excess agents on which rapid nucleation could occur.

The advantages of this precipitation method are well recognized in metallurgy. Grain refinement is largely by nucleation catalysts. Ti and Zr nucleate Al. Castings with fine grain size are desirable. This furnishes super plasticity for example. Precipitation from homogeneous solution gives purer, better crystallites. There is no local supersaturation. The rate of nucleation (as opposed to rate of grain growth) is faster. Fewer dendrites form and less solid strain is incurred during the solidification.

The goal sought in this experiment is to achieve metals and alloys with enhanced or unique properties through a better understanding of the nucleation process. This will be reached by pursuing the following scientific and technical objectives:

1. Determine nucleation kinetics for metals and alloys as a function of supercooling.
2. Determine interfacial energies by application of nucleation theory to the kinetic data.
3. Use data to test and extend homogeneous nucleation theory.
4. Define techniques and parameters necessary for producing new materials with enhanced properties through zero-g processing.

#### 7.13.2 Verification Requirements

Verification of the process parameters and material characteristics calls for the following:

1. Experimental conditions with different size of samples. Vonnegut showed that homogeneous conditions are approached by using small samples.
2. Experimental conditions with various degrees of supercooling - hence various times for solidification.
3. Experimental conditions at moderate, then high temperatures to permit orderly growth of the technology.

All experiments are to be performed at the lowest g-level commensurate with the flight hardware. Hence two levels of verification may be identified.

Level I. Three sample sizes of material melting at moderate temperature nucleated with moderate intensity supercooling,  $\approx 10^1$  degrees Centigrade.

Level II. Three sample sizes of material melting at elevated temperature nucleated after supercooling by  $\approx 10^2$  degrees Centigrade.

### 7.13.3 Materials

The materials for initial experiments are selected for their capability to provide the greatest quantity of information regarding characteristic differences when processed under conditions of one-g and zero-g. Hence it is desirable that the materials for early experiments will have been well studied at one-g so that evaluations and comparisons with low-g tests can be readily made.

The two materials selected, Sn and Ge have low and moderately high melting points respectively. Germanium has application in electronics and has optical properties which can aid in studying its structure. The candidate materials selected are listed below with some of their properties.

M.Pt. ° C	d	Interfacial Energy (s-1) erg/cm <sup>2</sup>	Heat of Fusion cal/g	Max Super- cooling, $\Delta T$ ° C	Cp cal/g/° C
Sn 232	7.29	54.5	14.2	118	0.074
Ge 934	5.32	181	10.1	227	0.053

### 7.13.4 to Definition of Samples, Experiments, and Experimental Facilities

#### 7.13.9

Further definitions are identical to Section 7.8 (Superconductors) for samples, processing, facilities and power. See sections 7.8.4 to 7.8.9. Also, the analytical techniques used to evaluate single crystals, sections 7.12.4 to 7.12.9, apply to homogeneous nucleation experiments with minor modifications.



It is noteworthy to remark here that the activities and equipment for homogeneous nucleation tests are intended to provide a sound theoretical and practical foundation for highly specific research experiments in the area of solid-liquid transition study and technology including crystallization, nucleation, glass production, grain growth and solidification from melts, in general. The particular requirements of each specific experiment will have to be defined in detail at the time it is proposed.

## 7.14 CONTAINERLESS ALLOYING

### 7.14.1 Process Definition and Objectives

The feasibility and potential of contact-free (containerless) melting of materials and liquid-state processing in zero-g has been discussed in a number of papers. Its basic acceptance as a promising application of zero-g processing is evidenced by (1) demonstration experiments scheduled for the initial Skylab mission (M-512); (2) inclusion in the "Blue Book" (Vol. VI) and (3) initiation of the development of a contact free heating and position control system. Containerless melting offers the following practical applications:

- (1) Processing of reactive metals
- (2) Processing of metals with extremely high melting temperatures
- (3) Research on nucleation and crystallization

Contact-free processing of reactive metals in zero-g (1) eliminates the terrestrial limitations in alloy preparation or alloy purity due to chemical reaction with the container material at liquid-state temperatures. It widens the use of liquid-state processes and permits the preparation of new alloys of such base metals as beryllium, zirconium or titanium. All these base metals have wide applications in chemical processing equipment. In addition, beryllium is attractive for electrical applications as its electrical conductivity at liquid nitrogen temperature is by an order of magnitude higher than copper or aluminum, zirconium is of importance in nuclear applications, and titanium has a wide use in high-performance aerospace structures and propulsion systems.

The problem in terrestrial alloying of materials exhibiting extremely high melting temperatures, such as tungsten or tantalum, is that there is often no container material available which withstands the temperature and at the same time does not react with alloying constituents. Similarly, terrestrial research on nucleation and crystal growth is hampered by the necessary presence of a solid support for the molten material which acts as a nucleation site, since it represents invariably the low end of the chemical gradient during solidification. In a contact-free liquid nucleation would be exclusively intrinsic

without any foreign interference. This may greatly enhance our understanding of the parameters governing nucleation and crystal growth and may lead to new applications in terrestrial as well as zero-g processing.

The objectives of containerless melting experiments in zero- or low-g are:

- (1) To verify the feasibility of producing new or improved alloys from reactive and refractory metals.
- (2) To obtain new or more accurate data on the mechanism of nucleation and crystal growth.

#### 7.14.2 Verification Requirements

The process postulates that the material is for some time, sufficient for alloying, in the liquid state and without contact to any other material. In earth-based (non-orbital) experiments it will be necessary to support the material during the heating period; upon melting it may either disengage from the support, or maintain only point-contact (sting support). Three processing methods have been considered:

- (1) The most attractive processing method is induction heating coupled with position control, using the free suspension system discussed in Sect. 7.15. In this case the sample is initially suspended on a thin wire of the sample material which melts shortly before sample melting, so that the entire liquid-state processing cycle including solidification is perfectly free of any contact. In view of the high temperatures involved, this method calls for an advanced version of the free suspension system. Since this has not become available within the timeframe of the initial low-g test program, other methods have to be considered.
- (2) One very simple method is radiation heating in an exothermic furnace of a sting-supported cylindrical, sample which, upon melting, forms a liquid sphere. Since cooling is slow, the sphere will remain in the liquid state for some time and will have to be sting-supported to stay in position. This suspension mode is illustrated in Fig. 6.2 (Sect. 6.1.1). The drawback of

this method is the chamber temperature limitation of exothermic furnaces.

- (3) A third experimental method is direct resistance heating of a cylindrical sample which, upon melting, forms one or more individual spheres. Since heating stops at the moment of sphere formation, solidification occurs within a few seconds - depending on the temperature level - and no position control or support is needed.

Of these three methods, exothermic heating has been discarded in view of the limited temperature capabilities which would permit only demonstration experiments. The selected methods (1) and (3) are adaptable to the high temperatures of practical refractory metals. They represent two distinct verification levels as follows:

Verification Level I: Short contact-free melting and alloying cycle, using a pre-mixed/compacted material sample and direct resistance heating.

Verification Level II: Extended contact-free melting and alloying cycle, using also a premixed/compacted material sample, induction heating and position control.

#### 7.14.3 Experimental Materials

The following table identifies candidate base materials and their applicable properties; specific alloys are not defined since the experimental requirements are adequately determined by the base materials. Of prime concern is the processing temperature which ranges from 1300 to 3200° C.

<u>Reactive Metals</u>	<u>Melting Temp ° C</u>	<u>Density gr/cm<sup>3</sup></u>	<u>Cp cal/gr</u>	<u>Lf cal/gr</u>
Beryllium	1,284	1.84	0.47	311
Titanium	1,660	4.5	0.12	104
Zirconium	1,860	6.44	0.078	53

#### Metals with High Melting Temperature

Chromium	1,850	6.92	0.13	70.1
Hafnium	2,230	13.1	0.035	34
Niobium	2,410	8.6	0.064	70

Metals with High Melting Temperature (Cont'd)

Molybdenum	2,620	6.1	0.061	69.4
Tantalum	2,850	16.6	0.033	41.5
Rhenium	3,167	21.0	0.032	424
Tungsten	3,410	18.85	0.032	60.1

For this evaluation and for initial experiments, Niobium has been selected as model material, since it represents a fairly high processing temperature (2500° C) which covers the majority of the candidate metals.

7.14.4 to 7.14.9 Definition of Samples, Experiments and Experiment Facilities.

All further definitions are identical to Sections 7.8 (Superconductors) for level I and 7.15 (Free Processing System) for level II, specific reference:

Verification Level I: Sections 7.8.4 to 7.8.9, all data applicable to level II superconductor experiments.

Verification Level II: Section 7.15.4 to 7.15.9 all data applicable to free processing experiments, phase III (2500° C).

## 7.15 FREE PROCESSING SYSTEM

### 7.15.1 Process Definition and Objectives

Contact-free processing is either the desired or the only effective method for all liquid-state and solidification processes where tooling contact would lead to chemical reactions or interferences with nucleation and crystal formation. It is mandatory for the following processes/materials:

- \* (1) Containerless Alloying
- \* (2) Superconductors - Dynamic mixing
- \* (3) High-temperature Metastable Alloys (Im-
- \* miscibles) - Dynamic Dispersion
- \* (4) Supercooling, Nucleation and Crystal Growth
- (5) Amorphous Oxides (glasses)

It is further required for specific techniques or objectives of:

- (6) Single crystal growth from the melt
- (7) Purification
- \* (8) Liquid/solid transition

All these processes are concerned with metals, with the exception of (5).

Those discussed in this report and identified by \*.

Contact-free processing requires a device which provides the following capabilities:

- (a) Position Control
- (b) Heating and Melting
- (c) Liquid Material Agitation

A fourth capability of contact-free shaping of liquids is not considered at this time.

A prototype contact-free processing system has been developed by General Electric, whose capability of position control (a) has been demonstrated in the form of levitation in one-g. Parameters for the control of position, temperature, liquid oscillation and material agitation in zero-g have been assessed theoretically. The exact definition of these parameters as a basis for the development of an operational system can, however, only

be obtained by experiments with a prototype system under zero- or low-g conditions.

The objectives of low-g experiments are:

- (1) Establishment of the control and systems design parameters for the capabilities (a, b, c) above.
- (2) Development of operational systems with increasing capabilities, commensurate with increasing processing experiment requirements.

#### 7.15.2 Verification and Systems Development Phases

For an effective experimental program, systems development experiments should be combined with processing experiments, using materials and processing conditions identified for applicable processing experiments, rather than model materials. It turns out that certain processing experiment levels agree well with the target capabilities (limitations) of the desirable steps in systems development. The following systems development phases provide an optimum for both purposes:

Phase I Prototype system - Evaluation of control- and design parameters

Phase II Development and fabrication of a processing system with the following characteristics:

1. Functional capabilities (a, b, c), above
2. Material (metals) mass equal to 1 cm diameter sphere.
3. Max temperature 1100° C
4. Single processing sample
5. Min. Weight/Volume Design

Phase III Processing system with the same characteristics as II, except for a temperature capability up to 2500° C.

Phase IV Operational system with the characteristics of III, except for larger material masses and multiple sample deployment.

Phase IV is beyond the timeframe base of this study and is not further considered. For the same reason, the processing of non-conducting materials is omitted, aside from the potential necessity of an entirely different systems concept.

### 7.15.3 Experimental Materials

Specific materials are identified in the discussion of the applicable processing experiments. They comprise the following material types and max processing temperatures:

	<u>Max. Processing Temp (° C)</u>		
Systems Type:	I	II	III
* Metastable Alloys		1100	3000
*Superconductors			2400
Free Alloying			2500
*Supercooling/Nucleation		1000	2500
Purification		1100	2500
Spheres (Al)	700		
Hollow Spheres		700	1600

Phase I experiments are limited to aluminum spheres, either solid (deployment and position control evaluation), or liquid if possible.

Initial phase I experiments are limited to the evaluation of position control and sample deployment techniques with solid Al-spheres. Subsequently it may include melting experiments and liquid spheres, either with Al, or a metal with a low latent heat of fusion per unit of volume, such as Mg ( $150 \text{ cal/cm}^3$  as compared with 250 for Al), Sb (104) or Bi (122).

The selection of specific materials for phase II and III experiments is determined by the scheduling of processing experiments. Pacing processing experiments are identified by asterisks in the listing of materials, above.

### 7.15.4 Material Quantity and Sample Size

The maximum material quantity for all (I, II, III)experiments is  $0.524 \text{ cm}^3$ , equivalent to a max. sphere diameter of 1 cm. The original sample configuration may be cylindrical, cubical or near-spherical with the following dimensions:



Cylindrical: 0.875 diam x 0.875  
Cubical: 0.808 x 0.808 x 0.808  
Spherical: Average diam 1 cm.

#### 7.15.5 Processing Phases

Individual processing phases may vary with the processing experiment requirements, but consist basically of:

- (1) Sample suspension
- (2) Coil cooling (and chamber pressurization, if applicable)
- (3) Heating to melting - position control
- (4) Heating to processing temperature and processing
- (5) Cooling through sample solidification
- (6) Sample recovery.

The basic process flow diagram is shown in Fig. 7.15-1.

#### 7.15.6 Low-g Experiment Requirements

##### 7.15.6.1 Low-g Time Requirements

For Phase I position control experiments with a solid sample, the desirable low-g time is 6-8 seconds and the minimum acceptable time 3 seconds. Experiments including melting require a min. low-g time of 8 seconds, predicated on the use of a material with a low heat of fusion per unit of volume, and preheating to a few degrees below the melting point.

For the evaluation of all systems capabilities in phase II and III experiments, a minimum low-g time of 40 seconds is required. Depending on the processing experiments to which the system is applied, the low-g time may be substantially longer and as defined in the concerned experiment discussions.

##### 7.15.6.2 g-Levels

Generally, the g-level should be as low as possible. For phase I experiments, a max. g-level of  $10^{-2}$  is acceptable. For phases II and III the g-level is determined by

the selected processing experiment requirements.

#### 7.15.7 Low-g Facilities and Experiments

This section is limited to rocket experiments (Level II and III), since drop tower/KC-135 experiments (Level I) are already in an advanced state of development.

The experiments have a dual purpose: (1) to verify the functional performance of the free suspension system and to obtain data for equipment optimization; (2) to perform contact-free processing experiments with various materials. The desirable low-g time is primarily determined by (2), yet varies with specific materials and processing temperatures. In view of the wide applications range, data are defined for a high operational envelope, or high sample temperatures and extended low-g processing times (Level III).

The maximum number of experiments per flight is first determined by apparatus space (axial height) experiments vs. space limitations and then checked against power and weight limitations.

7.15.7.1 Payload Space Limitations. The height of the individual processing module, as defined in 7.15.8, is 18 cm and the available apparatus space 80 cm, accommodating four experiments per flight and an (optional) central argon supply system.

7.15.7.2 Power Requirements. Since the prime objective of the experiments is to manipulate a molten sample, the power assessment is based on the input required to maintain maximum sample temperature. For a 1 cm diameter sample and a processing temperature of 2,000°C the following data apply:

Radiation loss (emissivity = 0.5)	250 watts
Input energy absorbed by sample (avg)	20%
Input required to maintain 2200° C	1250 watts
Power required for manipulation	100 watts
Total	1350 watts

For heating to processing temperature within less than 30 seconds, a somewhat higher input with a peak rate of 1800 watts will be required. The average input over the total processing period, including position control during the cooling period, has been computed to 1600 watts.

7.15.7.3 Power and Time Limitations. The postulated extended processing time calls, at least, for Trajectory B (max. standard WSMR) with a low-g time of 390 sec. For this trajectory, the payload weight capacity of RR-1 is insufficient (85 kg) and the use of RR-4 (Aerobee 200 - 125 kg) is mandatory. With the four experiments performed in sequence, roughly 90 sec are available for each experiment. This low-g time is perfectly adequate for all materials processing and equipment data requirements. The remaining 30 seconds are retained as a contingency for radiation cooling from lower processing temperatures.

The total processing power consumption can be computed on the basis of a constant input rate of 1600 watts over a total active processing time of 360 seconds. The resulting processing power consumption is 160 wh. Support equipment (recorder etc) requires app. 150 watts over 900 seconds or 38 wh. The total consumption of 198 wh is well within the 2-battery support module supply of 220 wh.

7.15.7.4 Weight Limitations. As shown in Sect. 7.15.8.4, a four-module apparatus is well within the weight capacity of RR-4/Traj. B. It leaves a contingency of 3 kg which may be used for optional equipment.

7.15.7.5 Experiment Definition. On the basis of the foregoing evaluation, rocket experiments are defined as follows:

Low-g Facility	RR-4 (Aerobee 200)
Trajectory	B (Max. Standard WSMR)
Total Low-g Time	390 Sec.
Number of Expts/Flight	4
Active Processing Time/Expt.	90 Sec.
Number of Samples/Expt.	1
Total Power Consumption	198 wh.

### 7.15.8 Apparatus and Payload Definition

7.15.8.1 Level I Experiment Equipment. Equipment for drop tower and/or KC-135 experiments is presently in the hardware stage (G. E. ) and is, therefore, not discussed.

7.15.8.2 Rocket Experiment Apparatus. As in other experiments, the modular apparatus design is preferable for the reasons stated in Sect. 3.6. One sample is processed per module. The modules are selfcontained, except for the high-frequency power supply which is provided by the support module. Major module components are:

- (1) Secondary power conditioning system
- (2) Control system
- (3) Experiment chamber with coil system
- (4) Coolant supply
- (5) (Camera-optional).

An independent module cooling system - rather than a central coolant supply - is preferable in view of coolant management considerations under low-g conditions. The basic processing assembly, consisting of components (1) to (3), above is a rectangular box, measuring app. 20 x 16 x 14 cm. Various arrangements of this unit and the coolant system have been evaluated. The most effective arrangement is illustrated in Fig. 7.15-2: The coolant tank consists of 1.75 helical turns of a 1 1/8" ID tubing, providing the required coolant capacity of 1 liter. Flexible connections from one end of the coil and from the pump at the other end minimize the transfer of mechanical vibrations. This design assures perfect coolant circulation. This assembly leave ample space for optional equipment such as a camera. Major module data are as follows:

Envelope Dimensions: 32 diam x 18 cm high.

Weights:

Processing Unit	11 kg
Cooling System	2.5 kg
Coolant	1 kg
Structures	<u>1</u> kg
Total	15.5 kg

Coolant Weight: The use of 1 liter water as coolant is based on the assumption that the total power input per module of 40 wh = 34, 000 cal has to be absorbed. The resulting max water temperature increase is 34° C.

7.15.8.3 Rocket Support Module. The support module includes the experiment power supply and primary power conditioning. Total power is 220 wh or app. 800 KW sec, with a max discharge rate of 3000 watts intermittent or 2500 watts continuous. Major envelope data are as follows (numbers in parenthesis identify components specified in Sect. 5.5.2 and Table 5-1):

Basic Structure (1, 2, 3)	33 kg
2 Batteries (4)	6 kg
Power Conditioning (5-A, B, C2)	12 kg
Sequencer, Recorder	4 kg
	<hr/>
Total Weight	55 kg
 Total Axial Height	 70 cm
Net Space for Apparatus	80 cm

7.15.8.4 Rocket Payload Assembly. A typical (dedicated) payload assembly, accommodating 4 processing modules, is shown in Fig. 7.15-3. The total payload weight and space data are as follows:

Payload Weight

4 Processing Modules	62 kg
Argon Supply System	5
Support Module, Net	<u>55 kg</u>
Total Payload	122 kg
 RR-4, Traj. B Payload	
Capacity	125 kg
Contingency	3 kg

### Payload Space (Axial Height)

Support Module	70 cm
4 Module Apparatus	72 cm
Argon Supply System	7 cm
Total Payload Height	149 cm
RR-4 Capacity	150 cm
Contingency	1 cm

#### 7.15.9 Experiment Performance

Since drop tower experiments are already in progress, performance is defined for rocket experiments only.

##### 7.15.9.1 Ground Operations at the launch site consist of:

- (1) Dry payload check-out
- (2) Charging with expendables
- (3) Second check-out (functional, measuring)
- (4) Vehicle Installation
- (5) Final check-out

7.15.9.2 Flight Operations. The sequencing of flight operations is pre-set and defined in the time diagram, Fig. 7.15-4. Experiments are carried out in sequence to preclude power supply interference.

##### 7.15.9.3 Post Flight Operations at the launch site consist of:

- (1) Payload Recovery
- (2) Removal of Processing Modules
- (3) Discharging of Expendables
- (4) Recovery of Samples, Recorder Tape and Telemetry Records for Evaluation

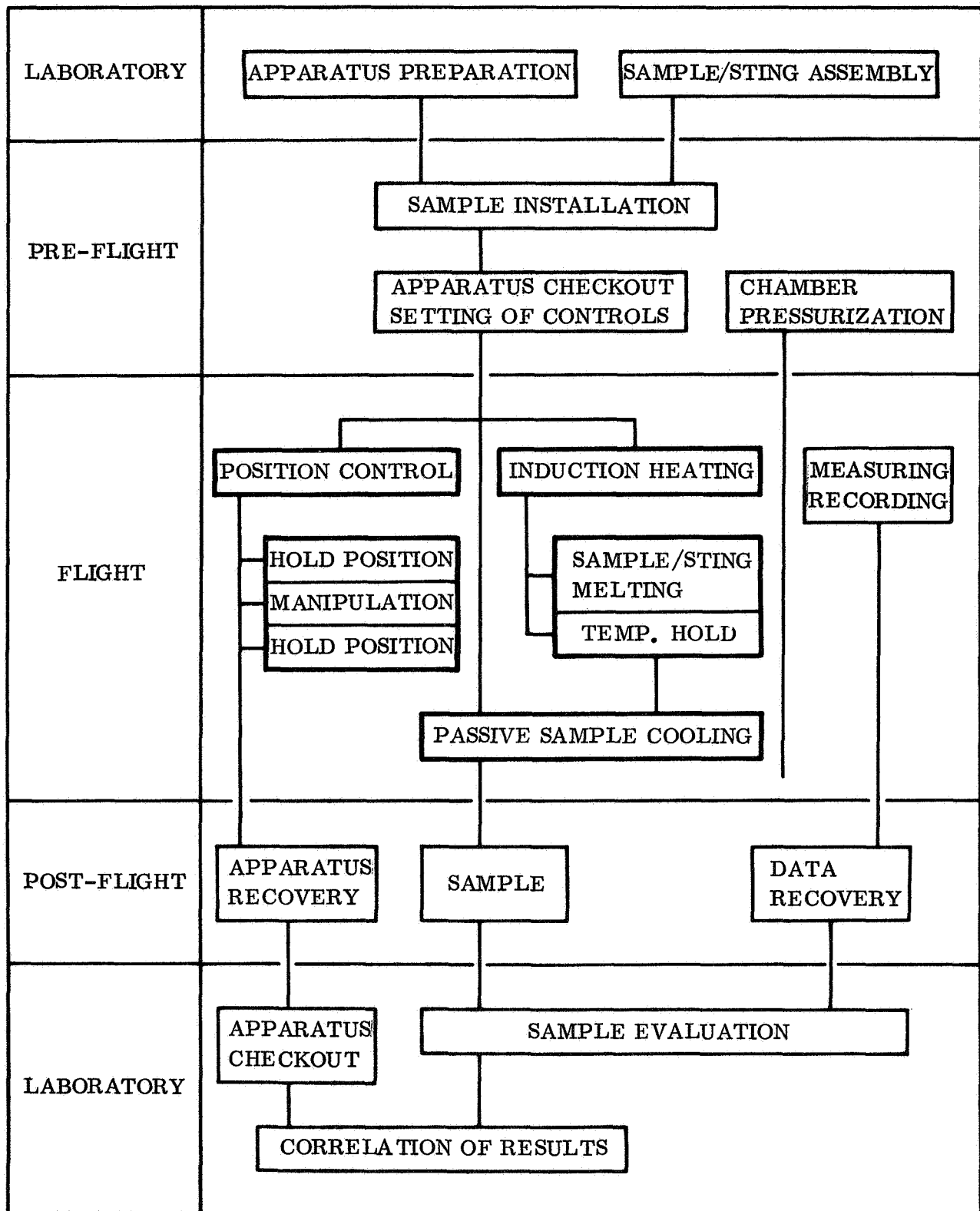
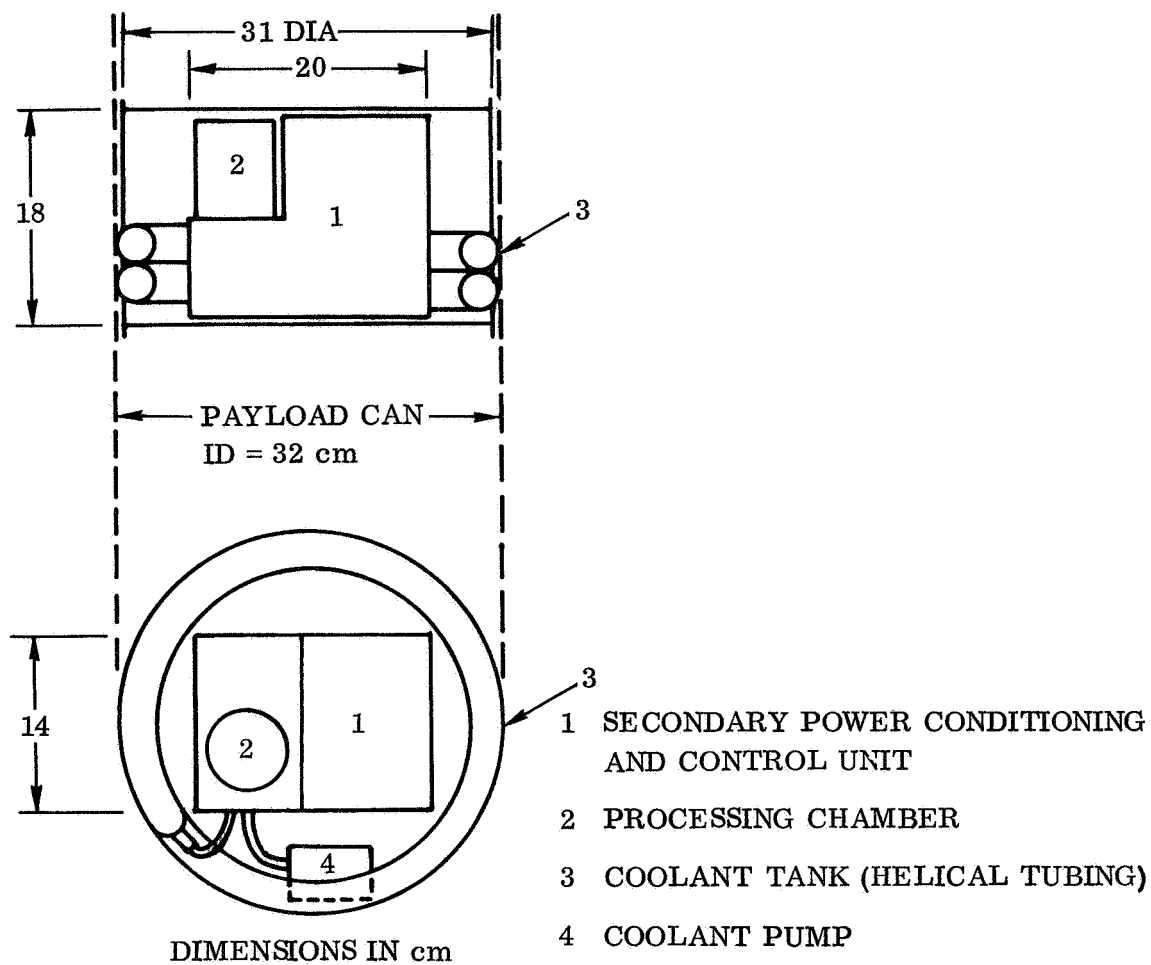


Figure 7.15-1. Process Flow Diagram - Free Processing Experiments



WEIGHT = 15.5 kg (INCLUDING COOLANT)

Figure 7.15-2. Free Processing Module



## ROCKET PAYLOAD ASSEMBLY

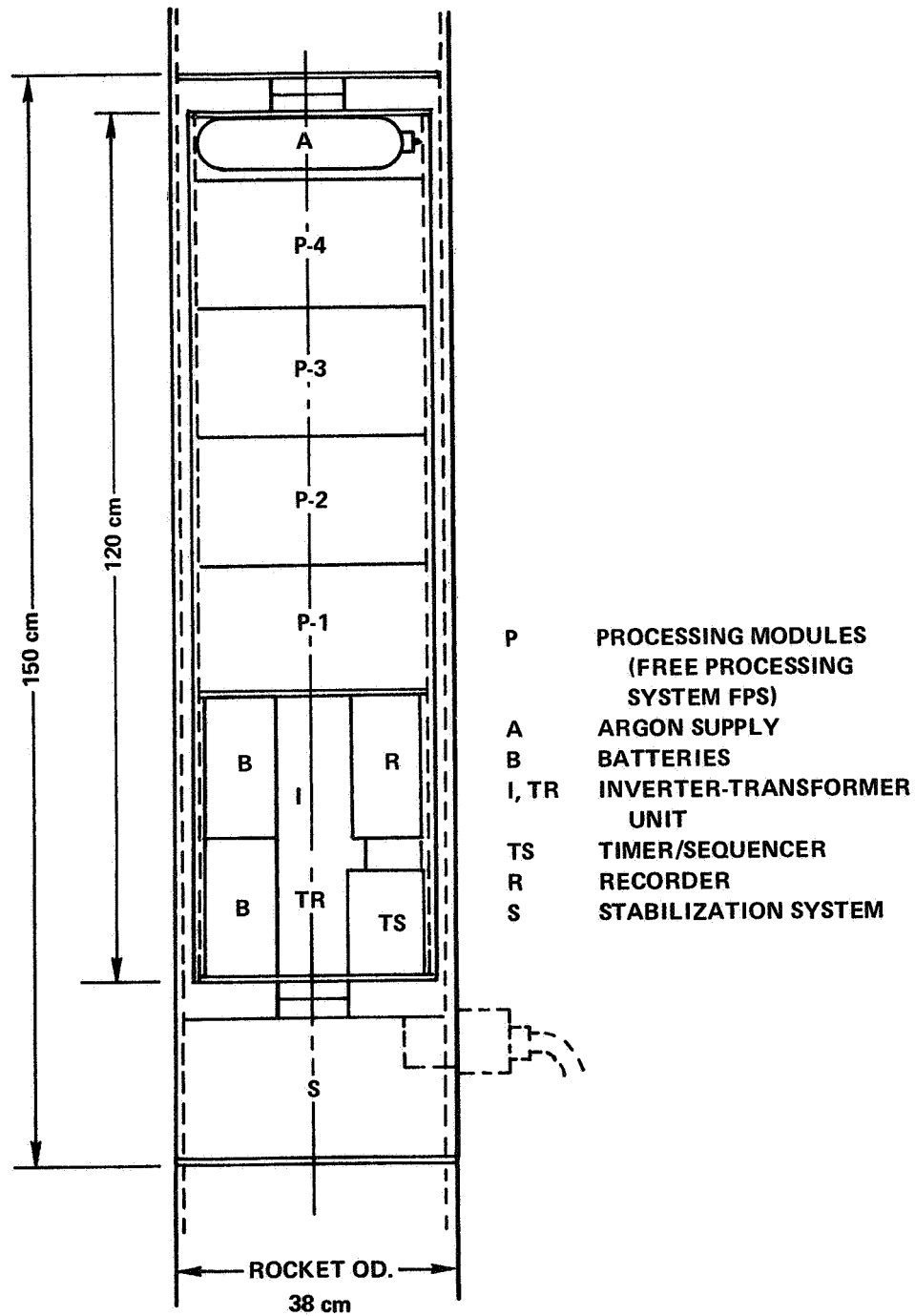


Figure 7.15-3. Payload Assembly — Free Processing System

# EXPERIMENT PROGRAM – TRAJECTORY B

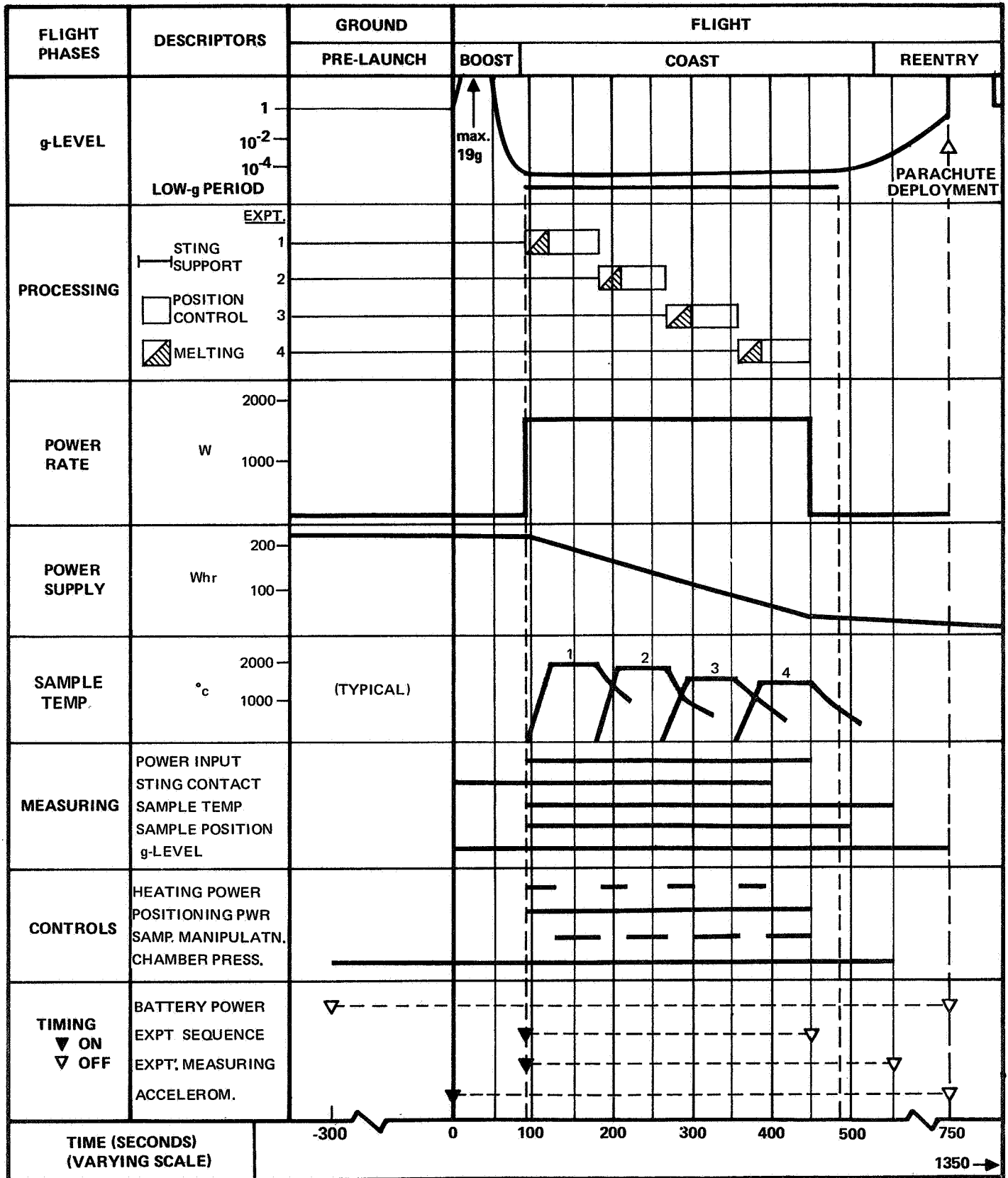


Figure 7.15-4. Free Processing System

## 7.16 LIQUID STATE FORMING (MEMBRANES)

The art of shaping materials in the liquid state, in the form of casting, is as old as man's knowledge of metals. It implies invariably two process phases: (a) liquid-state forming and (b) solidification for shape retention. Process performance in one-g, referred to as casting, calls for a mold or, at least, a supporting surface. In zero-g the basic process phases - liquid-state forming and solidification - are unchanged, except that we no longer need an enclosure or support surface, eliminating completely the countless problems associated with tooling contact.

Liquid-state forming in zero-g may be divided into four process categories.

- (1) Shaping by intrinsic material forces (surface tension) without any tooling contact
- (2) Shaping by surface tension and a minimum or pointwise contact, either with a tool (sting) or at a solid/liquid interface
- (3) Shaping by a controlled interaction between surface tension and liquid/solid transition.
- (4) Contact-free shaping by means of induced forces, such as electromagnetic or centrifugal forces.

The unique behavior of liquids in zero-g and its application to forming processes has been extensively discussed in a number of reports and papers. A variety of processes and products have been defined conceptually, such as perfect spheres and hollow spheres, metallic or nonmetallic ellipsoids, membranes, filaments. In contrast to all other processes and experiments which deal with materials and, consequently, exhibit a high commonality in equipment requirements, liquid-state forming calls for highly specialized devices for each individual product type. The only exception is the apparatus for contact-free shaping with electromagnetic forces (4), which will be an outgrowth of the free processing system (Sect. 7.15). All other shaping processes combine heating and cooling devices with mechanical systems.

In line with the point-design concept, one typical process - the drawing of membranes - has been selected for this evaluation. The choice was based on three considerations:

- (1) Uniqueness to zero-g conditions, (2) high interest for practical applications, (3) typical

equipment requirements (space, weight and power requirements representative of most other shaping processes), (4) drop tower experiments in progress will provide experience and data for extended-time experiments.

#### 7.16.1 Process Definition and Objectives

Under terrestrial conditions the thickness (or, rather, thinness) and flatness of membranes is highly limited by gravity-induced effects, particularly during the lifting of the solidified film from the surface which supported it during liquid-state forming. In the zero-g process, the membrane is "drawn" directly from the liquid without tooling contact except for edge guides. The shaping process is analogous to the conventional drawing of a solid material, where the deformation is forced by a nozzle. In the case of liquid-state forming in zero-g the nozzle is replaced by the interaction between surface tension and the tension generated by the drawing rate; the film thickness is further determined by the solidification rate. As it is the prime objective to produce ultra-thin membranes, drawing and solidification rates are very high. This is favorable to continuous processing, in which the controlled rpm of the take-up spool determines the drawing rate. The objective of the process is to produce continuous lengths of ultra-thin membranes from metallic and nonmetallic materials, for applications in composites, electroactive devices and chemical processing, including semi-permeable membranes.

#### 7.16.2 Verification Requirements

Experiments call for a highly specialized apparatus with provisions for melting of the supply material and a mechanical system which maintains the delicate balance between surface tension, drawing rate and solidification rate. Two levels of process verification are indicated:

Verification Level I: Process demonstration and evaluation of process details with single sample.

Verification Level II: Demonstration and optimization of the continuous process.

### 7.16.3 Experimental Materials

Since the prime objective of this experiment is process development, materials play - at least in the initial development phases - a subordinate role. Tin and copper have been tentatively selected as working materials for process development. The choice of a material with a low and a higher melting temperature is designed to obtain data for two distinctly different conditions during the membrane formation process (passive solidification rate).

### 7.16.4 Sample Definition

For Verification Level I, the sample is produced with a fixed-size draw-frame, approximately 5 cm wide and 20 cm long, yielding a max size sample of the same dimensions. The sample thickness cannot be defined, since the attainable minimum thickness is one of the objectives of the experiments.

For the continuous process of Verification Level II, the draw frame is replaced by either edge guides or coiled foil strips. The sample width is also 5 cm; the target sample length is 200 cm (coiled).

Since only a small fraction of the supply material is used for membrane formation, the required material quantity is identical for both experiment levels and amounts to 12 cm<sup>3</sup>. For Sn and Cu, the corresponding weights and heats of fusion are as follows:

	<u>Sn</u>	<u>Cu</u>
Quantity (gr)	88	107
Heat of Fusion (cal)	1260	5250

### 7.16.5 Experimental Process Definition

The preparation, performance and evaluation of low-g experiments consists of the following major phases:

#### (1) Preparation

- (a) "Dry" functional check-out of the drawing apparatus
- (b) Setting of controls - ready for operation

- (c) Charging with material
- (d) Apparatus Installation
- (2) Performance
  - (a) Heating and melting of the supply material
  - (b) Drawing under controlled temperature and atmosphere conditions
  - (c) Mechanical closing of the supply container
- (3) Evaluation
  - (a) Recovery of apparatus, sample and test recordings
  - (b) Sample Evaluation
  - (c) Correlation of sample characteristics with measured processing parameters.

#### 7.16.6 Low-g Test Requirements

7.16.6.1 g -Level. In all experiments, only the drawing phase (2b), above, is g-sensitive. For Level I a max g-level of  $10^{-3}$  g is desirable,  $10^{-2}$  g acceptable. For Level II, the g-level should not exceed  $10^{-5}$  g.

7.16.6.2 The Low-g Time required for levels I and II and the selected model materials are as follows:

Level I	Sn	3 sec
	Cu	2 sec
Level II	Sn	60 sec
	Cu	40 sec

For Level II the max time of 60 sec is adopted as point design value.

#### 7.16.7 Low-g Facilities and Experiments

The low-g processing times defined above show that Level I experiments can be carried out in the drop tower (or KC-135), whereas Level II experiments fall into the typical low-g time regime of research rockets. Since drop tower experiments are already in progress, they are

not further defined and the remaining discussion is confined to rocket experiments. The objective of the following evaluation is to define the max number of experiments which can be performed in one flight (dedicated mission), using equipment data specified in 7.16.8.

#### 7.16.7.1 Payload Space Limitations (in axial height)

Required Space for 6 Modules	75 cm
Available Apparatus Space	90 cm
Contingency	15 cm

#### 7.16.7.2 Payload Weight Limitations

6-Module Apparatus Weight	67 kg
Support Module	45 kg
Total Weight	112 kg
RR-1/Traj. A Capability	130 kg
Contingency	18 kg

7.16.7.3 Power and Time Requirements. Since the prime power consuming phase of melting is carried with ground power prior to launch, battery supply is required only for maintaining the melt through the boost phase, for temperature control of the processing chamber and for support equipment. The max (Cu) power requirements for one and for six experiments (modules) are as follows:

<u>Power Rate (w)</u>	<u>1 Expt.</u>	<u>6 Expts.</u>
Heating (Temp Hold)	200 w	1200 w
Support Equipment	120 w	200 w
Total Power Rate	320w	<u>1300 w</u>
<u>Energy Consumption (wh)</u>		
Temp. Hold (0-90 sec)	5 wh	30 wh
Drawing (90 - 150 sec)	3.4 wh	21 wh
Support (0-600 sec)	20 wh	34 wh
Total Consumption	29 wh	<u>85 wh</u>

It can be seen that for six concurrent experiments the max power rate of 1300 w and the total consumption of 85 wh are well within the capacity of one battery with a max discharge rate of 1500 w and a total stored power of 110 wh for a dedicated payload of 6 concurrent experiment the required low-g time is only 60 seconds. The modest time and power requirements make this experiment attractive for mixed-experiment payloads.

7.16.7.4 Experiment Definition. The low-g time of 60 seconds is way beyond the capabilities of drop towers or aircraft; it can, however be satisfied with the minimum rocket capability. The data for a dedicated payload are as follows:

Low-g Facility	RR-1
Trajectory	A (Min. WSMR)
Number of Expts./Flight	6
Total low-g Processing Time	240 sec
Max. g-level	$10^{-5}$ g
6-Module Apparatus Weight	67 kg
Total Energy Consumption	85 wh
Max. Power Rate	1300 w

#### 7.16.8 Apparatus and Payload Definition

7.16.8.1 The Apparatus design for rocket experiments is based on the experiences obtained in the construction of a drop tower apparatus under contract NAS8-28056.

Each module is of rectangular configuration; major data are:

Length and Width	14 x 12 cm
Height	25 cm
Weight	10 kg
Max. Power Rate	800 w
Max. Energy Consumption	10 wh



Two apparatus assemblies can be conveniently installed side-by-side in all types of research rockets.

7.16.8.2 Support Module. In view of the modest power requirements, the minimum support module outfitting (power directly from battery) is adequate for a dedicated payload. Weight and space requirements are (numbers in parentheses identify components specified in Sect. 5.2.2 and Table 5-1):

Basic Structure (1, 2, 3)	33 kg
1 Battery (4)	3 kg
Power Distribution & Controls	3 kg
Sequencer, Recorder, Misc. (6, 7, 8)	<u>6 kg</u>
Total Weight	45 kg
Net Space for Processing Modules (axial Height)	90 cm

7.16.8.3 Payload Assembly. The assembly of a dedicated payload is shown in Fig. 7.16-1. Major data are as follows:

Payload Weight

Support Module	45 kg
6 Processing Modules	60 kg
Gas Supply System	<u>7 kg</u>
Total Weight	112 kg

Payload Space (Axial Weight)

Support Module	60 cm
Apparatus	<u>75 cm</u>
Total Height	135 cm

### Payload Power

Total Energy Supply	110 wh
Consumption, 6 Modules	60 wh
Consumption Support Equipm.	25 wh
Total Payload Consumption	85 wh
Contingency	25 wh
Max Discharge Rate	1200 w

### 7.16.9 Experiment Performance

7.16.9.1 Ground Operations at the launch site after routine payload check-outs and charging with expendables (water, gas) consist of:

- (1) Ground activation of payload systems at -1000 sec.
- (2) Concurrent preheating of processing modules from -900 to 0 seconds.

7.16.9.2 Flight Operations. The sequencing of flight operations is pre-set and defined in the Time Diagram, Fig. 7.16-2.

7.16.9.3 Post-Flight Operations after payload recovery consist of:

- (1) Removal of processing modules from payload assembly.
- (2) Removal of samples from each module.
- (3) Recovery of flight recordings.
- (4) Evaluation of samples and flight recordings.

# ROCKET PAYLOAD ASSEMBLY

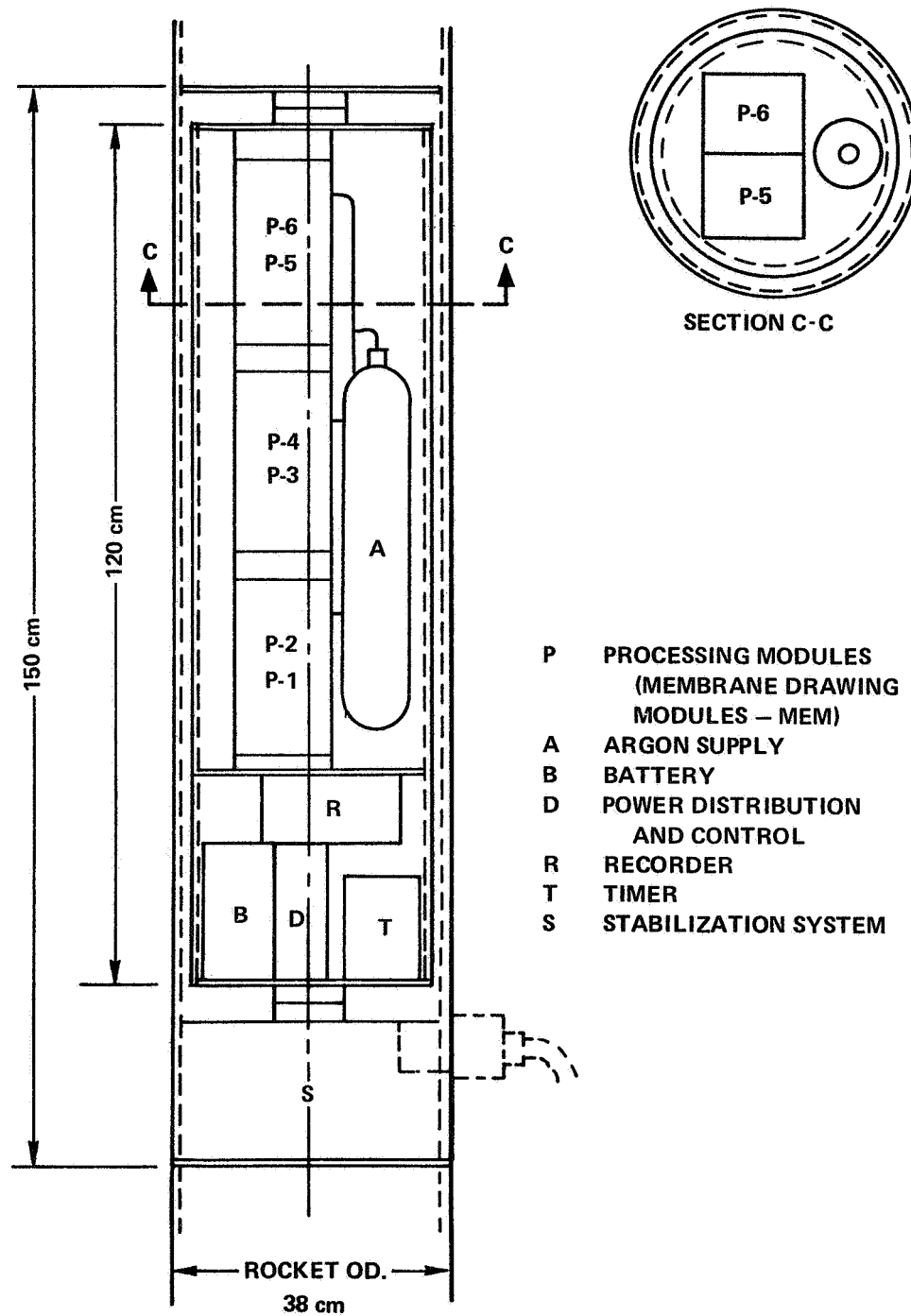


Figure 7.16-1. Payload Assembly — Liquid State Forming  
(Membrane Drawing)

# EXPERIMENT PROGRAM – TRAJECTORY A

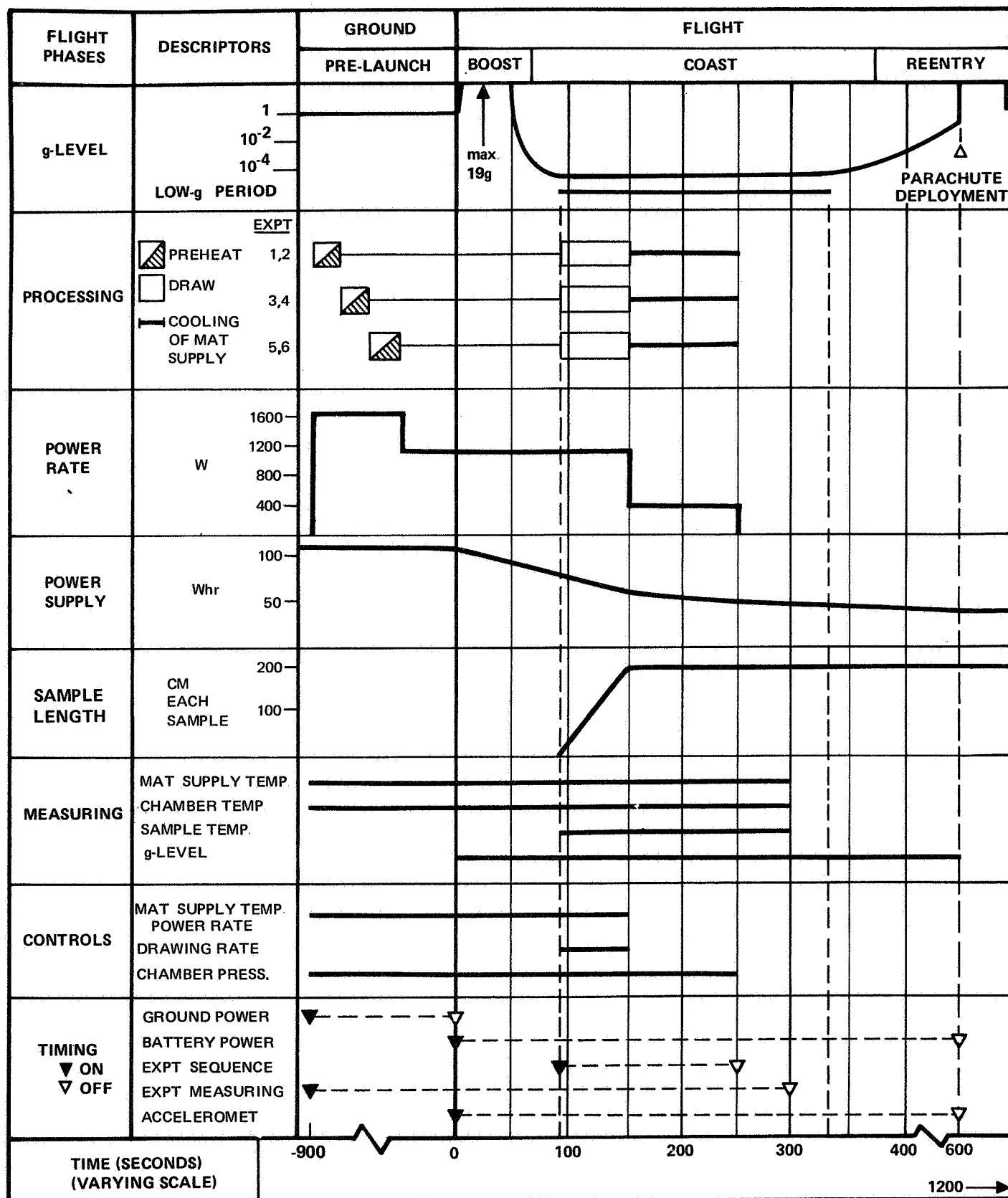


Figure 7.16-2. Drawing of Membranes

## 7.17 OXIDE GLASSES

In the earlier phases of this study (Table 3, Section 2 and Interim Report No. 1 of Aug. 1, 1972) the preparation of new glasses was excluded from consideration for initial extended-low-g experiments in spite of the high applications potential and the uniqueness of the process to the zero-g environment. This original exclusion was based on the rationale that the leadtimes for the highly specialized processing equipment exceeded the timeframe of an initial low-g experiment program. Since then, glasses have been reinstated for the following reasons:

- (1) In the course of the study, experimental techniques have been defined which can be adapted to the processing of glasses.
- (2) Several low-melting glasses of high practical interest have been defined by the USRA Advisory Committee on glasses and some of its members.
- (3) The high technological significance of low-g produced glasses justifies accelerated developmental efforts.

The basic principle of the low-g preparation of glasses is contact-free solidification of the molten material which is expected to inhibit nucleation and crystal formation, resulting in an amorphous product. This is unfeasible in one-g, due to the necessity of a container which acts as a nucleation site.

Two types of glasses have been selected for experiments: (1) oxide glasses with high processing temperatures, discussed in this section, (2) chalcogenide glasses with low melting temperatures evaluated in Section 7.18. The authors are aware that, by precise definition the term "chalcogenide" includes oxides since it comprises certain compounds of all elements of Group VI. The introduced designation of the two glass categories, though not accurate, appeared acceptable as matter of practical convenience.

### 7.17.1 Objectives and Process Definition

The general objective of this process is to produce unique amorphous oxides for optical and - by secondary processing - semi-conductor applications. The specific objectives of low-g

experiments are (a) feasibility demonstration, (b) to produce sample quantities which permit the measuring of the essential product properties and (c) to obtain data on the processing of non-conductors as a basis for the development of larger-scale processing facilities.

The process consists of heating to a temperature at which all intrinsic nuclei are dissolved, followed by contact-free solidification. Two verification levels have been defined:

Verification Level I: Preparation of small quantities in the order of  $0.05 \text{ cm}^3$  by minimum-contact melting and zero-contact solidification.

Verification Level II: Preparation of larger quantities of  $1 \text{ cm}^3$  or more by contact-free melting and solidification.

Initial experiments, discussed in this section, are limited to verification level I.

The following evaluation of these experiments is based primarily on the extensive developmental work on oxide glasses carried out since 1968 by R. Happe of NAR. Many valuable suggestions have been made by Mr. Happe which have since been incorporated in this study. It may also be stated at this point, that the resulting definition of experiments and techniques is in full agreement with experimental concepts proposed earlier by Mr. Happe.

#### 7.17.2 Verification Requirements

Process verification calls for (1) an experimental processing technique which, even though not necessarily ideal, permits a measurable distinction of the product properties achieved in one-g and low-g and (2) a sample quantity adequate for the measurement of these properties. In the ideal case, the process calls for contact-free heating to a temperature in the liquid state at which complete solution of all intrinsic nuclei is achieved, maintaining this temperature for an extended period, followed by contact-free (radiation) cooling through solidification. In the experimental process certain deviations from the ideal conditions will be necessary in view of the problems associated with the contract-free heating of non-conductors and the high processing temperatures. The following modifications have been

accepted: (a) during melting the sample material will not be absolutely contact-free, but will be supported by solid sample material (semi-free), (b) the hold-time at the max temperature is only in the order of several seconds. However, practically ideal processing conditions will be maintained during the most critical cooling and solidification phase.

A material quantity of  $0.05 \text{ cm}^3$  or equal to a sphere of app. 0.5 cm diam. has been found adequate for the evaluation of the crystallinity of the material. It also will permit a prediction, yet no accurate measurement, of optical properties.

### 7.17.3 Experimental Materials

The following list of candidate experiment materials and the related data have been furnished by Mr. R. Happe of NAR. For each material, three temperatures are defined: (1) the melting temperature  $T_m$  (even though there is in most cases no discrete melting point), (2) the processing temperature  $T_{max}$  and (3) the glass transition temperature  $T_g$  at which the amorphous solidification is completed, or below which the material is no longer sensitive to contact. Materials are listed in the order of descending processing temperature  $T_{max}$ .

<u>Material</u>	<u><math>T_m</math> (<math>^{\circ}\text{C}</math>)</u>	<u><math>T_{max}</math> (<math>^{\circ}\text{C}</math>)</u>	<u><math>T_g</math> (<math>^{\circ}\text{C}</math>)</u>
$\text{Y}_2\text{O}_3$	2410	2600	1520
$\text{Gd}_2\text{O}_3$	2330	2530	1470
$\text{La}_2\text{O}_3$	2200	2400	1380
$\text{Al}_2\text{O}_3 + 20 \text{ w/o SiO}_2$	2050	2250	1190
$\text{Ta}_2\text{O}_5 + 10 \text{ w/o CaO}$	1870	2070	1160
$\text{Nb}_2\text{O}_5 + 15 \text{ w/o CaO}$	1490	1700	936

Other properties of significance for experiment design are electrical conductivity, thermal conductivity and emissivity. Accurate data are not available and their determination will be one of the initial tasks of experiment development.

In this process group, low-g experiments should be adaptable to a wide variety of materials. The definition of experiment requirements is, therefore, based on the entire

envelope of characteristics represented by the list of candidate materials, rather than one specific material. The following materials data have been adopted as baseline for the subsequent evaluation:

Max. processing temperature	1700 - 2600 <sup>o</sup> C
Min. temperature range	
( $\Delta T$ ) of contact-free cooling from $T_{\max}$	900 <sup>o</sup> C
Emissivity in this range	0.6 to 0.9
Lower limit of electrically conductive temperature regime	1500 - 1800 <sup>o</sup> C

#### 7.17.4 Experimental Process Definition

For this process the sequence of sub-section 7.  $\times$  .4 (sample definition) and 7.  $\times$  .5 (experimental process definition) has been reversed, since the sample configuration depends upon the selected processing technique.

7.17.4.1 Process Selection. The critical processing requirements for the achievement of an amorphous product have been defined in the discussion of the verification requirements, Sect. 7.17.2. They call for a processing technique which provides the following major characteristics:

- (1) Heating to extremely high temperatures up to 2600<sup>o</sup>C.
- (2) Contact-free cooling to the glass transition temperature  $T_g$  or, as defined in Sect. 7.17.3, over a  $\Delta T$  of 900<sup>o</sup>C.

In addition one should list a third requirement, typical of all low-g experimentation, namely a high degree of simplicity and reliability with regard to equipment and experiment performance.

No elaboration is made of the advantages and disadvantages of the various techniques which have been analyzed for this application. In most cases the overriding shortcoming was excessive equipment requirements. The net result of this analysis was the adoption



of a dual radiation/resistance heating technique and contact-free solidification without position control.

**7.17.4.2 Heating.** For other processes associated with similar extreme temperatures two heating methods were adopted: Direct resistance heating (7.8, 7.10, 7.14) and induction heating (7.14, 7.15). A prerequisite of both methods is an electrically conductive material. In the present case we deal with materials which are classified as nonconductors. However, the conductivity increases with temperature and reaches between 1500 and 1800°C an order of magnitude adequate - even though not very efficient - for heating by direct resistance or induction. A dual heating method was, therefore, adopted in which the sample is first heated to 1500 - 1800°C by radiation, followed by direct resistance heating to the desired max. processing temperature  $T_{\max}$ . Direct resistance was selected for the second heating phase in view of its superiority over induction heating with regard to equipment requirements.

Another reason for the selection of this method is the equipment commonality with other experiments, which reduces development cost and time: It consists essentially of a combination of the radiation heating system illustrated in Fig. 7.12-1/2 and applied in experiments 7.7, 7.12 and 7.13, and the direct resistance heating system illustrated in Fig. 6-12 and applied in experiments 7.8, 7.10 and 7.14. The combined system is described in Sect. 7.17.8 and illustrated in Fig. 7.17-3.

**7.17.4.3 Cooling.** The postulation of zero-contact during solidification from  $T_{\max}$  to  $T_g$  makes radiation cooling imperative. In the concerned high temperature regime the cooling rate is extremely high which, in turn, eliminates the need for position control. Whether the cooling rate has any effect upon amorphous solidification is unknown. If necessary, the cooling time could be prolonged by simultaneous radiation heating which could be accomplished with the devised heating system. Rapid cooling as it occurs naturally may, however, be beneficial as it may minimize the formation of intrinsic nucleation sites.

**7.17.4.4 Atmosphere Control.** The ideal environment for the high-temperature processing of oxides is an oxygen-rich atmosphere at a pressure in the order of 1-2 atm. This

presents severe experimental difficulties, as it would preclude the use of metallic components in the processing chamber. Chamber pressurization with argon, though not perfect, was considered acceptable, as it fulfills at least some of the environmental requirements and, at the same time, provides oxidation protection.

**7.17.4.5 Processing Phases.** The selected experimental processing method comprises the following major steps (g-sensitive phases are identified by asterisks):

- (1) Material and sample preparation
- (2) Preparation of the sample assembly and installation in the apparatus
- (3) Pressurization of the processing chamber
- (4) Heating to  $T_{\max}$ 
  - (4a) Radiation heating to 1500-1800<sup>o</sup>C
  - (\*) (4b) Direct resistance heating to  $T_{\max}$
- (\*) (5) Cooling from  $T_{\max}$  to below  $T_g$
- (6) Final cooling and sample deposition in processing chamber
- (7) Sample recovery and evaluation.

The sequence of operations is identified in more detail in the process flow diagram, Fig. 7.17-1.

#### **7.17.5 Sample Configuration**

The configuration of the sample and sample assembly is determined by the heating and cooling techniques described in the preceding section 7.17.4. Referring to the sample assembly in Fig. 7.17-3, heating starts with the activation of both transformer systems T-1 and T-2. The tungsten heating element RH, which is part of the single secondary turn of T-1, arrives quickly at a temperature of 2000<sup>o</sup>C, heating the sample S to a temperature of 1500-1700<sup>o</sup>C within app. 20 seconds. As the sample temperature and, consequently its electrical conductivity increase, the direct resistance heating, powered by T-2, becomes gradually effective. As soon as the current output of T-2 has reached a certain magnitude, T-1 is deactivated and further sample heating to  $T_{\max}$  is accomplished solely

by direct resistance heating. Upon melting, the formation of a single liquid sphere is assured by proper sample configuration ( $L/D = 4$ ). Arcing at the moment of sphere detachment is prevented by proper combination of voltage and gas pressure. In view of the high sphere temperature and the fact that the radiation heating element has cooled-off in the meantime, radiation cooling is very effective and the temperature  $T_G$  at which the sphere material becomes insensitive to contact will be reached within 1 to 3 seconds for the candidate materials listed in 7.17.3 and the sample size listed below. During this short, yet critical interval and at a g-level of not more than  $10^{-5}g$  the sphere should essentially stay in place, eliminating the need for position control. At higher g-levels it may take as long as 15 seconds before the sphere comes in contact with parts of the sample assembly; after such period, the sample material is, for all practical purposes, fully solidified.

To facilitate this process, tungsten sleeves are placed over the sample ends as shown in Fig. 7.17-3. Their purpose is twofold: (1) to compensate for the lower conductivity of the cooler sample ends during the transition from radiation to resistance heating and (2) to define the exact "gage" length necessary for a clean sphere formation. The data of the actual sample are as follows:

Diameter	0.4 cm
Total length	5.2 cm
Melting (gage) length	1.6 cm
Total molten volume	$0.2 \text{ cm}^3$
Diameter of sphere	0.45 cm
Sphere volume	$0.047 \text{ cm}^3$

#### 7.17.6 Low-g Processing Requirements

7.17.6.1 Low-g Time. The duration of the complete processing cycle is 50 seconds (max). It is composed of the following intervals:

(1) Pure radiation heating	22 sec
(2) Radiation and resistance heating	10 sec

- |                                           |        |
|-------------------------------------------|--------|
| (3) Pure resistance heating               | 13 sec |
| (4) Cooling to $T_g - 300^\circ \text{C}$ | 5 sec  |

The correlation of these periods with power inputs and sample temperature are illustrated in Fig. 17.7-2. Low-g conditions are required for two reasons and in two periods: (1) during the viscous part of the heating profile and the solid-liquid transition in order to maintain sample shape and to achieve acceleration-free sphere formation, (2) during solidification to a point below the glass transition temperature  $T_g$  to assure contact-free sphere suspension. This time range is represented by periods (2) to (4), above which plans the required low-g processing time at 28 seconds.

7.17.6.2 g-Level. The g-level is dictated by the initial solidification phase (4, above) during which the sample should remain free of any contact. To assure minimum displacement of the sphere formed and released upon reaching  $T_{\text{max}}$ , without the aid of a position control system, g-forces should not exceed a level of  $10^{-5}$  g.

#### 7.17.7 Low-g Facilities and Experiments

The required low-g time of 28 seconds far exceeds the capabilities of drop towers and aircraft, yet can be conveniently provided by research rockets. The specific rocket type and trajectory are determined by the most efficient flight utilization, represented by the number of individual experiments for a dedicated payload. The possible number of experiments, in turn, is determined by weight, power and/or time limitations.

7.17.7.1 Weight Limitations. According to the payload weight data of 7.17.8.4, a maximum of five (5) processing modules can be accommodated on either RR-1 or 4. The weight comparison is as follows:

4-Experiment Payload	126 kg
RR-1-Traject. A	130 kg
RR-4-Traject. B	125 kg

The slight overweight of 1 kg for RR-4-B is acceptable. If necessary, it can be absorbed by

the 2-kg contingency provided for in the support module. A modification of the trajectory is avoided, since this would interfere with the composition of mixed payloads (Section 8.3, 8.4).

**7.17.7.2 Low-g Time Limitations.** The minimum low-g time for the individual experiment has been defined in 7.17.6.1 as 50 sec. It is desirable to perform the 5 experiments defined above in sequence with a total low-g time of 250 sec. This can be easily accommodated in RR-4-B (390 sec). While it is marginal for RR-1-A (243 sec), it could likewise be accommodated by starting the first experiment 10 sec. prior to the nominal low-g period or at +80 flight seconds.

In view of the possibility that the second heating phase requires more than the theoretically computed time, and in view of the desirability of an extended low-g cooling period, RR-4/Trajectory B has been selected which permits an increase of the low-g time for the individual experiment to 60 seconds, which is the maximum required under most unfavorable conditions. The total low-g time for a dedicated payload is then 300 sec. or from +90 to +390 flight seconds.

**7.17.7.3 Power Limitations.** Since the experiments are performed in sequence, the max. power rate is that of the individual experiment. According to Fig. 7.17-2 the power peak amounts to 1200 watts which is well within the max. battery discharge capability of 1500 watts. Power consumption is also no problem as evidenced by the following data:

Single experiment	11 Wh
5-Expt. payload	55 Wh
Support systems (900 sec $\times$ 100 W)	25 Wh
Total payload consumption	80 Wh
Stored power (1 battery)	110 Wh
Reserve	30 Wh

**7.17.7.4 Experiment Definition.** Major experiment specifications for oxide glasses are summarized as follows:

Low-g facility	RR Class 4
Trajectory	B (max standard WSMR)
Max. number of expts/flight	5
Low-g time/expt	60 sec
Total low-g time	300 sec
Max power rate	1200 w
Power consumption	
Single expt (net)	11 Wh
Dedicated payload (incl. support systems)	80 Wh

#### 7.17.8 Apparatus and Payload Definition

The payload consists of the apparatus, comprising up to 5 processing modules with a central gas supply system, and the support module.

7.17.8.1 Processing Module. The dual heating capability required for experimental processing technique described in Sect. 7.17.5 is provided by a processing module consisting of two transformer units and a processing chamber. It is essentially a modified version of the direct-resistance heating module defined in Section 6.2.3 and Fig. 6-12, introducing a second transformer unit. The processing chamber with the dual heating system is shown in detail in Fig. 7.17-3. A functional description of the heating system was presented in Section 7.17.4 in connection with the definition of the sample assembly. The module assembly is illustrated in the insert of Fig. 7.17-3. The significant module data are as follows:

Envelope dimensions (cm)	29 × 12 × 14 high
Chamber interior (cm)	6 × 6 × 9 high
Weight	14 kg
Max power rating	2 × 800 watts

### Typical transformer outputs

Transformer T-1	1V/750 amps
Transformer T-2	25-40 V/30-20 amps

7.17.8.2 The apparatus consists of several (5 max for dedicated payload) processing modules and a central argon supply system; the argon pressure bottle is arranged in the available payload space alongside the processing module stack. Apparatus data for a dedicated payload are:

Number of modules	5
Apparatus height	70 cm
Apparatus weight	
5 modules	70 kg
Argon system	5.5 kg
Total	75.5 kg

7.17.8.3 Support Module. The basic design of the support module is described in Section 5.0. The specific type required for oxide glass experiments is characterized by a power conditioning system with two inverters and no transformer. Major components and data are: (Numbers in parenthesis identify components specified in Table 501):

Stabilization system and basic structure (1-3)	33 kg
1 Battery (4)	3 kg
Power distribution (5A)	2.5
2 Inverters (5B)	6
Timer and recorder (6, 7)	4 kg
Contingency	2 kg
	<hr/>
	50.5 kg
Axial height	60 cm
Stored power	110 Wh
Max discharge rate	1500 Watts

7.17.8.4 Payload Assembly. The layout of the dedicated payload assembly is shown in Fig. 7.17-4. Payload weight and height are as follows:

	<u>Weight (kg)</u>	<u>Height (cm)</u>
5-Module Apparatus	75.5	70
Support Module	<u>50.5</u>	<u>60</u>
Payload	126.0	130
RR-4 - Traj. B Capacity	125.0	150
Contingency	(-1)	20

The 1 kg overweight can be absorbed either by the 2-kg support module contingency or by a slight modification of trajectory B (time contingency = 90 sec)

#### 7.17.9 Experiment Performance

This section is confined to the experiment performance operations and does not include sample preparation and evaluation.

##### 7.17.9.1 Ground Operations at the launch site:

- (1) Apparatus check-out
- (2) Installation of sample assemblies
- (3) Final check-out of systems/controls
- (4) Activation of chamber atmosphere control at -300 sec.

Since there is ample battery reserve, all pre-launch check-outs may be carried out with board power.

7.17.9.2 Flight Operations are detailed in the time diagram, Fig. 7.17-5. Since there is no preheating or terminal cooling, processing of all samples is completed within the low-g period (+90 to +390sec). Only atmosphere control and support systems are operated prior and beyond this period.

7.17.9.3 Post-Flight Operations comprise payload recovery and the removal of samples and flight recorder tapes for evaluation.



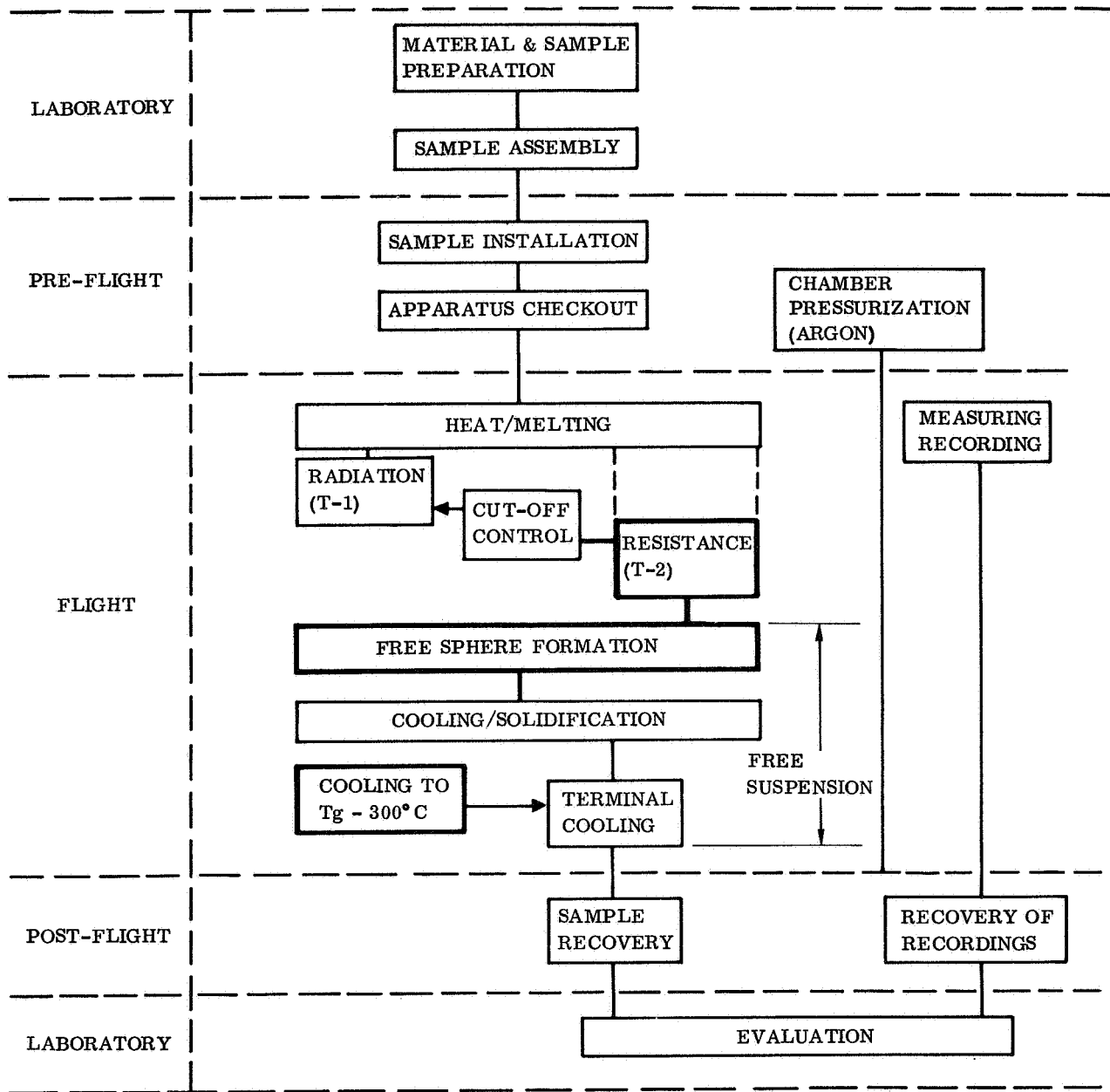


Figure 7.17-1. Process Flow Diagram - Oxide Glass Experiments

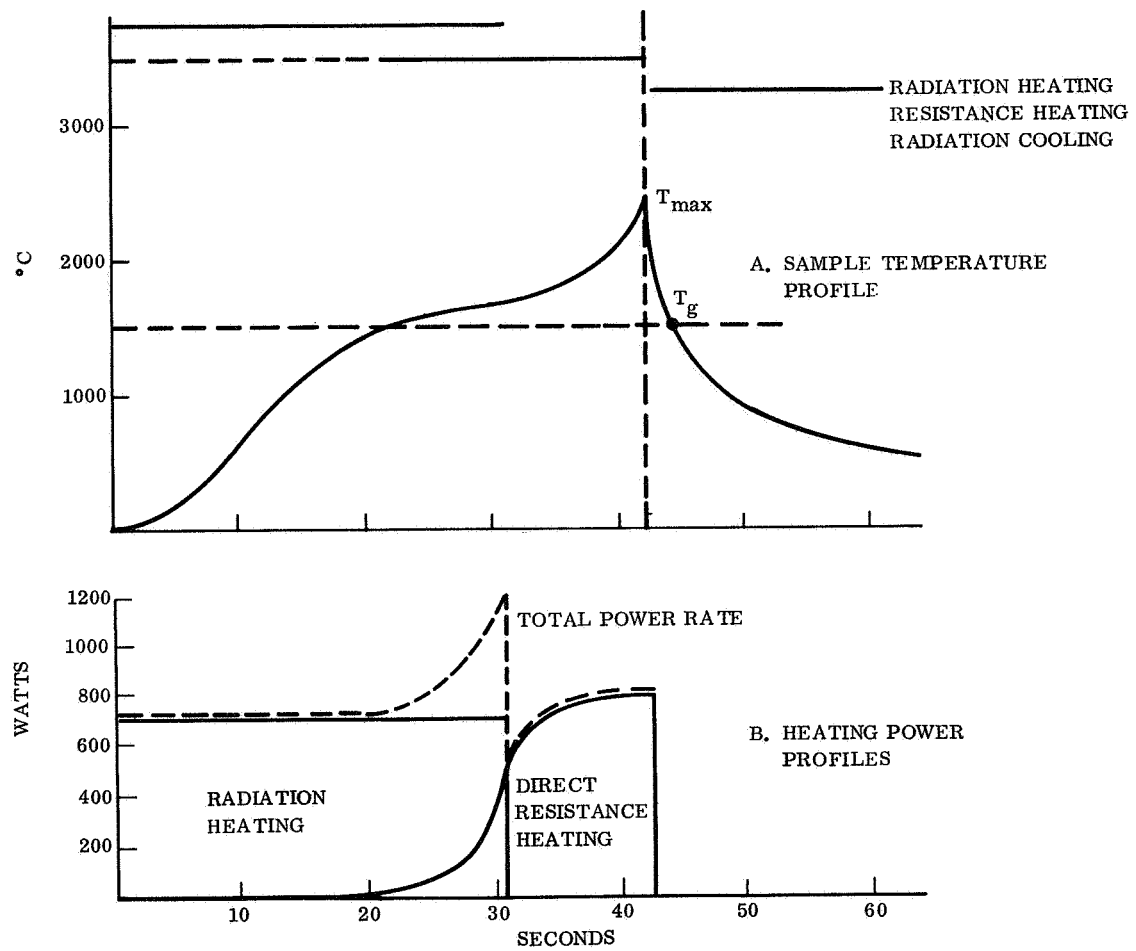
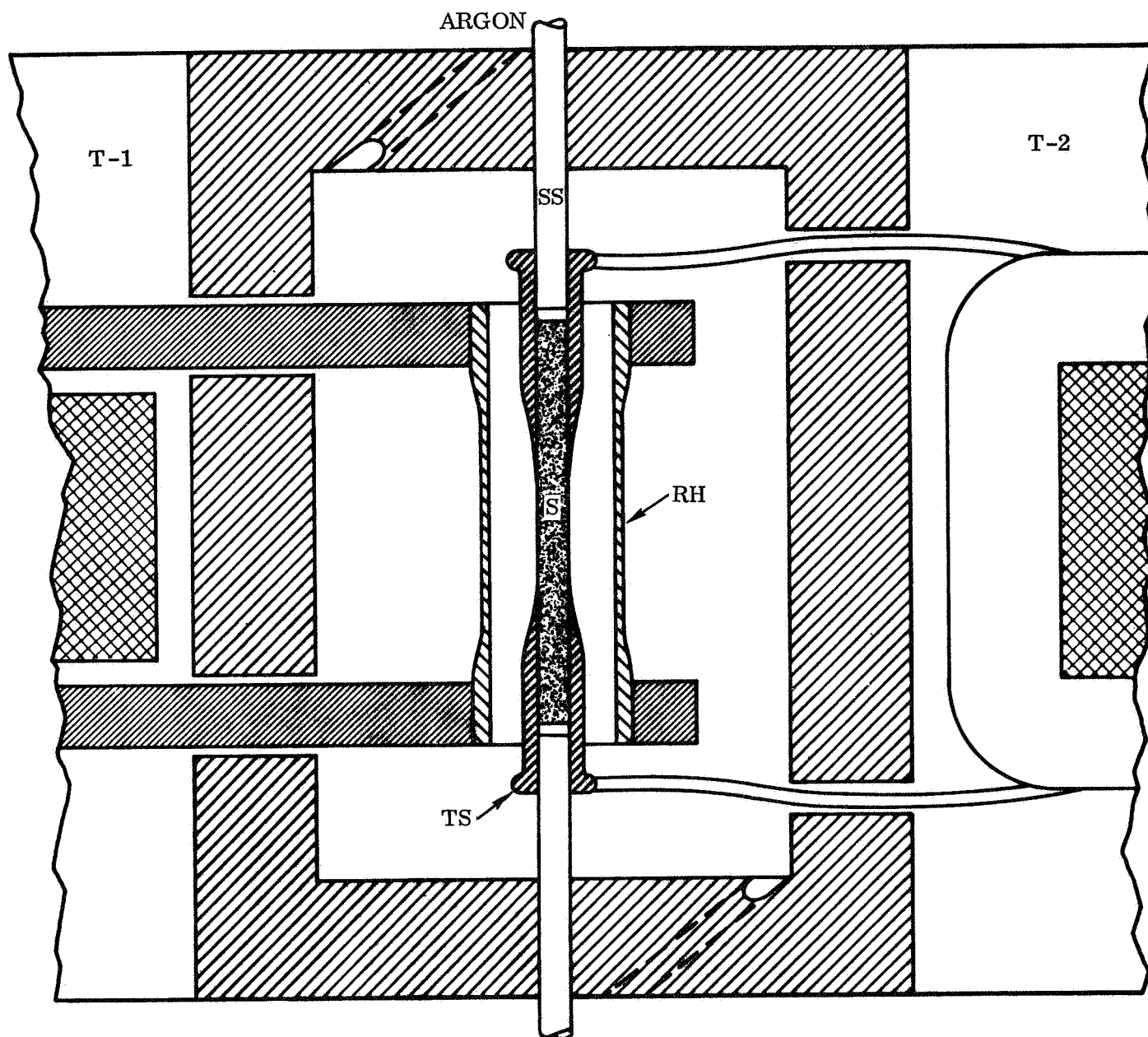


Figure 7.17- 2 Sample Temperature and Power Profiles for Oxide Glass Experiments



- S SAMPLE  
 TS TUNGSTEN SLEEVE  
 SS SAMPLE SUPPORT  
 RH RADIATION HEATER (W)  
 T-1 TRANSFORMER 1V-200 AMP.  
 T-2 TRANSFORMER 30V-25 AMP.

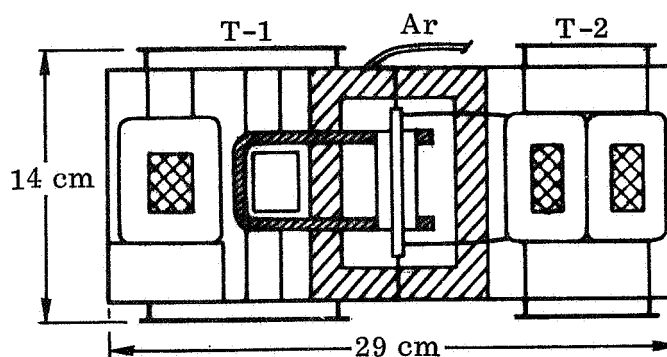


Figure 7.17-3. Dual Heating Module for High Temperature Glasses

# ROCKET PAYLOAD ASSEMBLY

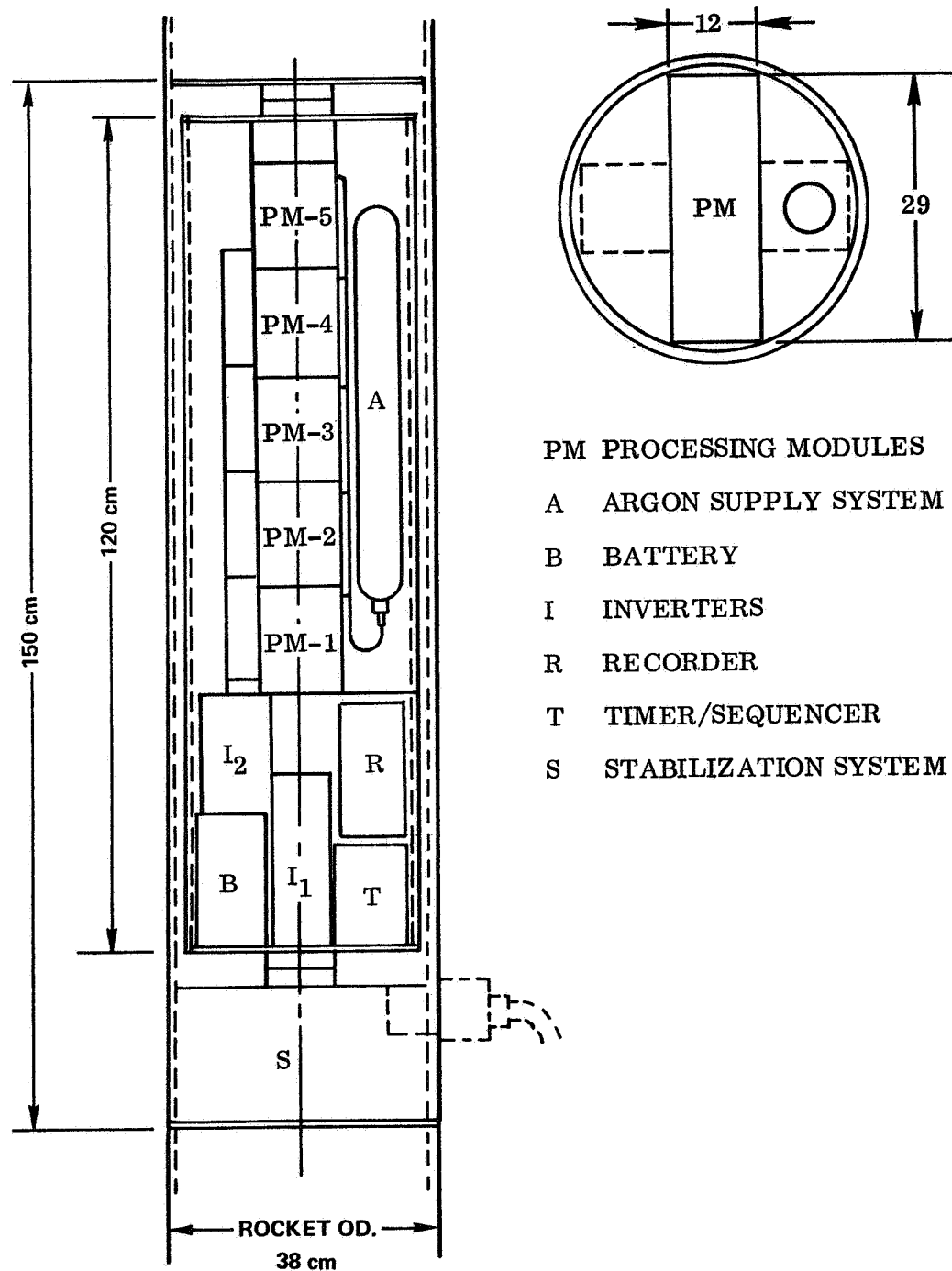


Figure 7.17-4. Payload Assembly - Oxide Glass Experiments  
 (Dedicated Flight)

# EXPERIMENT PROGRAM – TRAJECTORY B

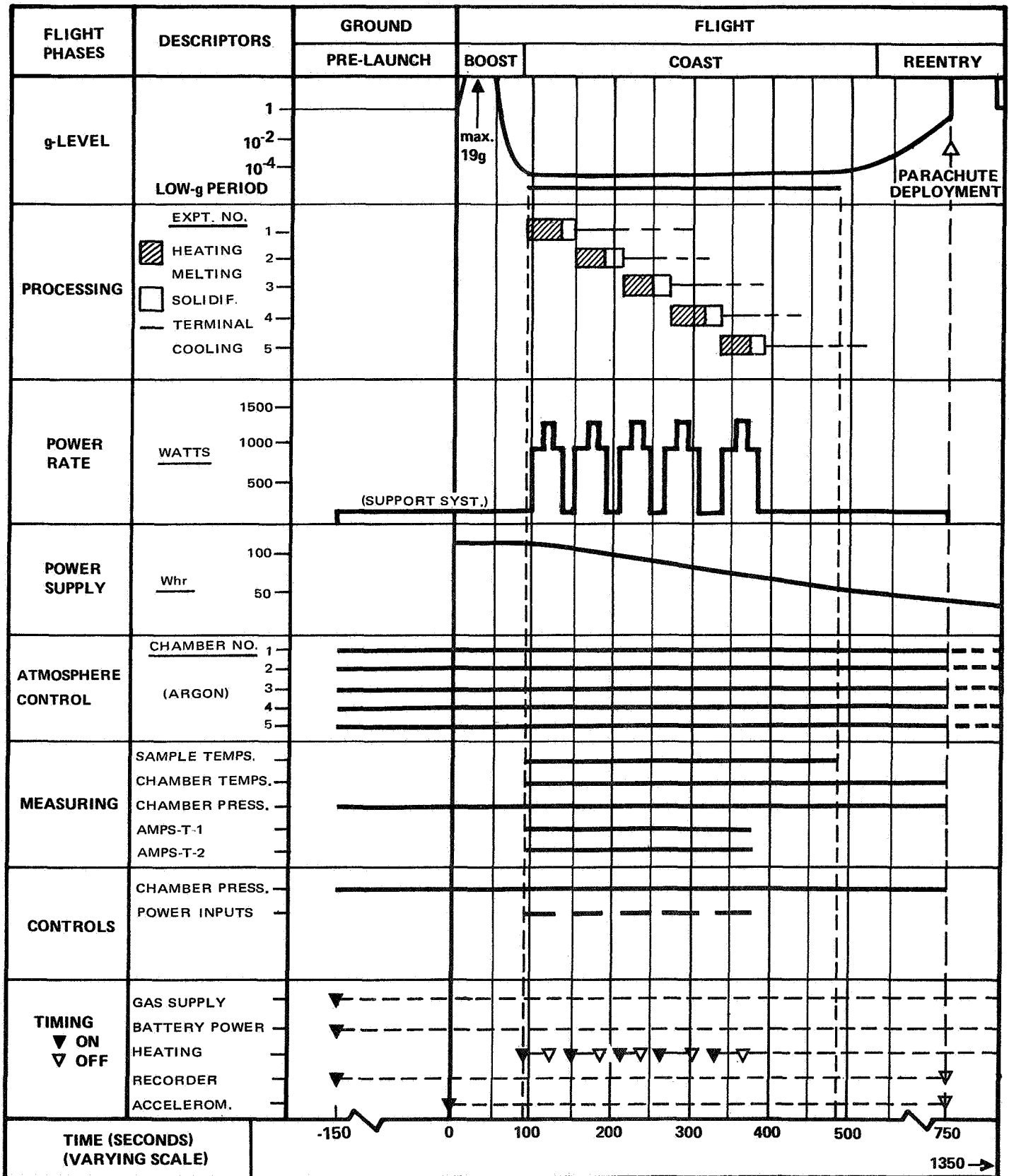


Figure 7.17-5. Experiment Program - Trajectory B (Oxide Glasses - Dedicated Payload)

## 7.18 CHALCOGENIDE GLASSES

This process/experiment group comprises glass materials which allow a significantly lower processing temperature than the oxide glasses discussed in the preceding section (7.17). Chalcogenides have been selected since they are of high interest for infrared optics, particularly low-absorption windows and lenses for  $\text{CO}_2$  lasers. Further, materials data required for this experiment evaluation were available from extensive work on chalcogenide glasses carried out by IITRI over the past two years. However, the defined experiments and apparatus are equally adaptable to experiments with crystalline glasses of high homogeneity ("glass ceramics") as they have been proposed by Grumman.

### 7.18.1 Process Definition and Objectives

The prime objective of low-g experiments is to produce amorphous chalcogenides of high purity. This is achieved by contact-free processing in the critical high-temperature regime. Containerless processing is expected to provide two effects: (1) amorphous solidification due to the absence of a nucleation site, (2) freedom from foreign metal contamination as it is encountered in terrestrial processing in oxide crucibles.

Specific experiment objectives are the exploration and definition of individual materials and processing parameters beneficial to amorphous solidification, such as variations in the material composition and in the time-temperature profile during high-temperature processing.

### 7.18.2 Verification Requirements

The requirements for the verification of the material characteristics implied by the stated objectives are:

- (1) Definition of promising material compositions and preparation of samples with compositional variations.
- (2) Provisions for contact-free and highly controllable heating and cooling of the sample material.

- (3) Provisions for sample position control during contact-free processing.
- (4) Evaluation of the product in the regard to material composition, microstructure and optical properties.

On the basis of the involved processing temperatures (7.18.3) and the availability of low-g facilities, three verification levels have been defined:

Verification Level I: Max processing temperature of 650° C and sample quantities up to 0.5 cm<sup>3</sup>, for exploratory experiments.

Verification Level II: Max processing temperature of 1150° C and sample quantities up to 0.5 cm<sup>3</sup>, for the preparation of practical materials .

Verification Level III: Larger material quantities for prototype applications.

The following evaluation is limited to verification levels I and II, or sample quantities up to 0.5 cm<sup>3</sup> (1 cm diam spheres).

### 7.18.3 Experiment Materials

Following suggestions made by Bill Crandall of IITRI, the following candidate materials have been selected. The listing defines composition, the max processing (equilibration) temperature, and the critical solidification temperature  $T_G$  below which the material becomes insensitive to contact:

<u>Verification Level I</u>	<u>T<sub>max</sub></u>	<u>T<sub>G</sub></u>
(1) As <sub>2</sub> S <sub>3</sub>	500° C	350° C
(2) As <sub>2</sub> Se <sub>3</sub>	500° C	350° C
(3) Ge <sub>28</sub> Sb <sub>12</sub> Se <sub>60</sub>	660° C	400° C
<u>Verification Level II</u>		
(4) Si <sub>6</sub> As <sub>4</sub> Te <sub>9</sub> Sb <sub>4</sub>	800° C	600° C
(5) Si <sub>45</sub> As <sub>25</sub> Te <sub>30</sub>	1050° C	900° C

Material (5) has been selected as model material as the applicable processing and equipment requirements cover all other materials. The specific composition of the model (point-design) material (5) is as follows:

15 SiTe<sub>2</sub>; 12.5 SiAs<sub>2</sub>; Balance Si, heavily doped with As.

#### 7.18.4 Sample Quantity and Configuration

Samples are prepared in near-spherical shape. Each sample is attached to a sting or filament of sufficient thickness to withstand accelerations up to 19g. The sting may be prepared from sample material or, preferably one of its constituents, such as Te or Se. Acoustic position control permits the processing of several samples in one chamber; in this case, the sample array, spaced in 1 or 2 wavelength distances, together with the suspension sting, are prepared as one unit.

The size of the individual spherical sample is between 0.6 and 1 cm (max) diameter. The definition of the accurate sphere diameter has to await development of, and laboratory experiments with, the acoustic position control system, identified conceptually in Sect. 7.18.8.

#### 7.18.5 Experimental Process Definition

The complete experiment, from sample preparation to the definition of the achieved product characteristics, consists of the following major phases:

- (1) Experiment Preparation
  - (11) Preparation of Sample Materials
  - (12) Preparation of Sample Assembly
  - (13) Sample Installation
- (2) Experiment Performance
  - (21) Acoustic Positioning (21-24)
  - (22) Heating to T max
  - (23) Hold at T max
  - (24) Cooling below T<sub>G</sub>
  - (25) Sample Recovery



(3) Sample Evaluation

(31) Microstructure (crystallinity, if any)

(32) Optical Properties

(33) Composition

(34) Correlation with processing parameters

Acoustic positioning is active throughout the entire thermal cycle, so that the individual samples are held in place immediately upon melting of the suspension filament or sting.

7.18.6 Low-g Test Requirements

Low-g conditions are required for the period of process phases 22 to 24, above. In view of the comparatively short heating times (see below), the entire processing cycle is carried out under low-g conditions and acoustic position control.

7.18.6.1 Processing Time Requirements. The thermal cycle consists of three phases: (1) heating to max (equilibration) temperature, (2) hold at this temperature and (3) cooling through the critical temperature  $T_G$ , below which the material becomes insensitive to contact and does no longer call for low-g conditions. The prime time is the hold at  $T_{max}$ , which should be as long as possible. While the heating and cooling periods (1, 3) vary for different materials, the time requirements are well represented by the following data:

<u>(Seconds)</u>	<u>Level I</u>	<u>Level II</u>
(1) Heating	40	50
(2) Hold at $T_{max}$	150	150
(3) Passive Cooling	50	30
Total	240	230

Heating is accomplished fast at a high power input rate to minimize the heat stored in the passively cooled chamber wall, essential for effective sample cooling by radiation. Heating data are defined in 7.18.8

7.18.6.2 G-Level. Since the sole purpose of low-g is maintaining contact-free sample suspension, and in view of the acoustic positioning, a g-level as high as  $10^{-3}$  g is acceptable.

#### 7.18.7 Low-g Facilities and Experiments.

The required total low-g time of 230-240 seconds places the experiments in the typical rocket regime. The performance of several experiments on one flight call for trajectory B with a max low-g time of 390 seconds, and for RR class 4 to meet the payload weight requirements. The objective of the following assessment is to define the max number of experiments which can be carried out on one (dedicated) flight.

7.18.7.1 Weight and Volume Limitations. In view of the comparatively low weight of the processing module (9.5 kg), the payload is primarily limited by space constraints. According to the data defined in 7.1.8.8, up to 4 modules (8 experiments) can be accommodated in the payload section occupying a total axial height of 80 cm.

7.18.7.2 Time and Energy Rate Limitations. It is now investigated whether a 4-module payload is feasible with regard to the available low-g time and the permissible power (discharge) rate. It can be accomplished if (1) the 8 experiments are evenly divided between Level I and II (4 experiments at a max temperature of 650° C and 4 at 1050° C), and (2) experiments are sequenced according to the following schedule (Expts. 1 to 4 represent Level I and 5 to 8 Level II):

<u>Low-g Time</u> (sec)	<u>Acoust.</u> P. C.(30W)	<u>Heating</u> (I-500W) (II-1000W)	<u>Temp Hold</u> (I-100W) (II-500W)	<u>Cooling</u> (0-W)	<u>Tot. Power*</u> <u>Rate (W)</u>
0-50	1-8	1, 2, 5, 6			3240
50-100	1-8	3, 7	1, 2, 5, 6		2940
100-150	1-8	8	1-3, 5-7		3040
150-200	1-8	4	1-3, 5-8		3040
200-250	1-8		3, 4, 7, 8	1, 2, 5, 6	1440
250-300	1-8		4, 8	3, 7	840
300-350	1-8		4	8	340
350-390	1-8			4	240

\* Data do not include payload support equipment (150W).

7.18.7.3 Power Requirements. The heating times and input rates used in the above schedule are based on thermodynamic calculations for the chamber configuration described in 7.18.8 and the use of tungsten heating filaments at a temperature of 2000° C. For the single experiment (chamber), the power requirements and related data have been defined as follows:

	<u>Level I</u>	<u>Level II</u>
Max Sample Temp	650° C	1150° C
Heating-Power Input	500 W	1000 W
Initial Heating Rate	40° C/sec	60° C/sec
Time to reach T max	40 sec	50 sec
Required to maintain T max	100 W	500 W

Total energy requirements (power consumption) have been computed as follows (all expt. requirements include operation of acoustic system):

<u>Watt Hours</u>	<u>Level I</u>	<u>Level II</u>
Single Experiment (1 chamber)	18	42
Single Module (2 chambers)	30	76
Support Systems (150W-900 Sec)	38	
4-Module Payload (incl support systems)	250	

7.18.7.4 Experiment Definition. According to this evaluation experiments and low-g facilities are defined as follows:

Low-g Facility	RR-4 (Aerobee 200)
Trajectory	B (Max. Standard WSMR)
Low-g Time/Expt.	240 sec
Low-g Time/8 Expt. Payload	390 sec
Max. Number per Flight of:	
Processing Modules	4
Experiments	8 (2 per module)
Samples	24 (3 per expt.)

#### Max Sample Temperature

Level I	650° C
---------	--------

Level II	1050° C
----------	---------

#### Total Power Requirements

Level I Experiment	18 wh
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Module	30 wh
--------	-------

Level II Experiment	42 wh
---------------------	-------

Module	76 wh
--------	-------

Dedicated Payload	250 wh
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#### 7.18.8 Apparatus and Payload Definition

The payload consists of the apparatus, comprising up to 4 processing modules, and the support module.

7.18.8.1 Processing Module. The processing module capable of heating to 1150° C and acoustic position control is shown in principle in Fig. 3.18-2. It consists of a central sound emitter and two processing chambers ("double-ender"). The samples are heated with tungsten filaments arranged in a circle of 6 cm diam. around the chamber axis. They are held in position after melting of the sting by standing acoustic waves generated between the acoustic emitter and reflector. (Chamber end-plates). High-purity argon atmosphere serves for both sound transmission and oxidation protection of the tungsten filaments. The tungsten filaments are dimensioned so as to provide an adequate radiation surface without undue obstruction to the sound passage. The surface of the cylindrical chamber walls is designed for an optimum compromise between the desirable acoustic and thermal properties.

The dimensions of the module are determined by the properties of the acoustic system. It is based on an operating frequency of 16 kHz. At this frequency, the wavelength in the argon atmosphere of the processing chamber and, consequently, the

sample spacing is app. 2 cm. The corresponding wavelength in the emitter material of 30 cm calls for an emitter length of 30 or 15 cm. In the module design of Fig. 7.18-2 the length of the half-wave emitter has been further reduced by proper configuration to 10 cm. The chamber dimensions of 14 cm x 12 cm ID are designed for an optimum combination of acoustic and thermal characteristics.

While this module is primarily designed for chalcogenic glasses, it may be adopted as a general purpose apparatus for all experiments which call for the combination of free material suspension and moderate temperature. Its major dimensional and operational data are summarized as follows:

Height x O. D.	40 x 14 cm
Chamber height x I. D.	14 x 12 cm
Number of proc. chambers	2
Acoustic frequency	16 kHz
Max. heating power rate	1000 W
Power rate/acoustic energy	60 W
Module weight	9.5 kg

7.18.8.2 The apparatus consists of the processing module(s), individual acoustic generators for each module and a central argon supply system. The acoustic energy generators are solid-state devices, measuring app. 16 x 10 x 4 cm each. Total apparatus space (in terms of axial height) and weight requirements for a dedicated payload are as follows:

	<u>h(cm)</u>	<u>Wt (kg)</u>
4 Processing Modules	80	38
4 Acoustic Generators	-	4
Argon Supply System	—	<u>6</u>
Total	80	48

7.18.8.3 Support Module. The apparatus calls for a support module with inverter and transformer (for heating, acoustic generators receive power directly from the battery). For the dedicated payload, the support module is defined as follows

(numbers in parenthesis identify components specified in Table 5-1):

Components and Weights

Basic Components (1-3)	33	kg
3 Batteries (4)	9	kg
Power Conditioning (5A, 5B, 5C-2)	12	kg
Timer/Sequencer (6)	1.5	kg
Recorder (7)	2.5	kg
Contingency (8)	<u>2.0</u>	<u>kg</u>
Total Weight	60	kg
Total Height	65	cm
Stored Power	330	wh
Max. Discharge Capacity	4200	w

7.18.8.4 Payload Assembly. The layout of the dedicated payload assembly is shown in Fig. 7.18-3. Payload weight and height are composed of the following:

	<u>Weight (kg)</u>	<u>Height (cm)</u>
4-Module Apparatus	48	80
Support Module	<u>60</u>	<u>65</u>
Total	108	145
RR4-Tr-B Capacity	125	150
Contingency	17	5

Total payload power requirements are 250 wh. The support module provides for three standard batteries with a total capacity of 330 wh, leaving ample reserve for check-out operations and contingencies.

7.18.9 Experiment Performance

This section is confined to the actual experiment and does not include sample and apparatus preparation or sample evaluation.

7.18.9.1 Ground Operations at the launch site consist of the following major steps:

- (1) Apparatus check-out (installed in payload), particularly with regard to precise frequency of acoustic systems.
- (2) Sample installation
- (3) Final systems and controls check-out
- (4) Activation of chamber atmosphere control at -300 sec.

Since there is adequate battery reserve, all pre-launch tests are carried out with board power.

7.18.9.2 Flight Operations are detailed in the time diagram, Fig. 7.18-4. Actual experiment performance starts at the beginning of the low-g period (+90 sec). While all thermal processing is essentially finished at the end of the low-g time (+480 sec), acoustic positioning remains active for another 100 sec to delay contact of the samples with the chamber wall.

7.18.9.3 Post-Flight Operations comprise apparatus recovery and the removal of samples and recordings for evaluation.

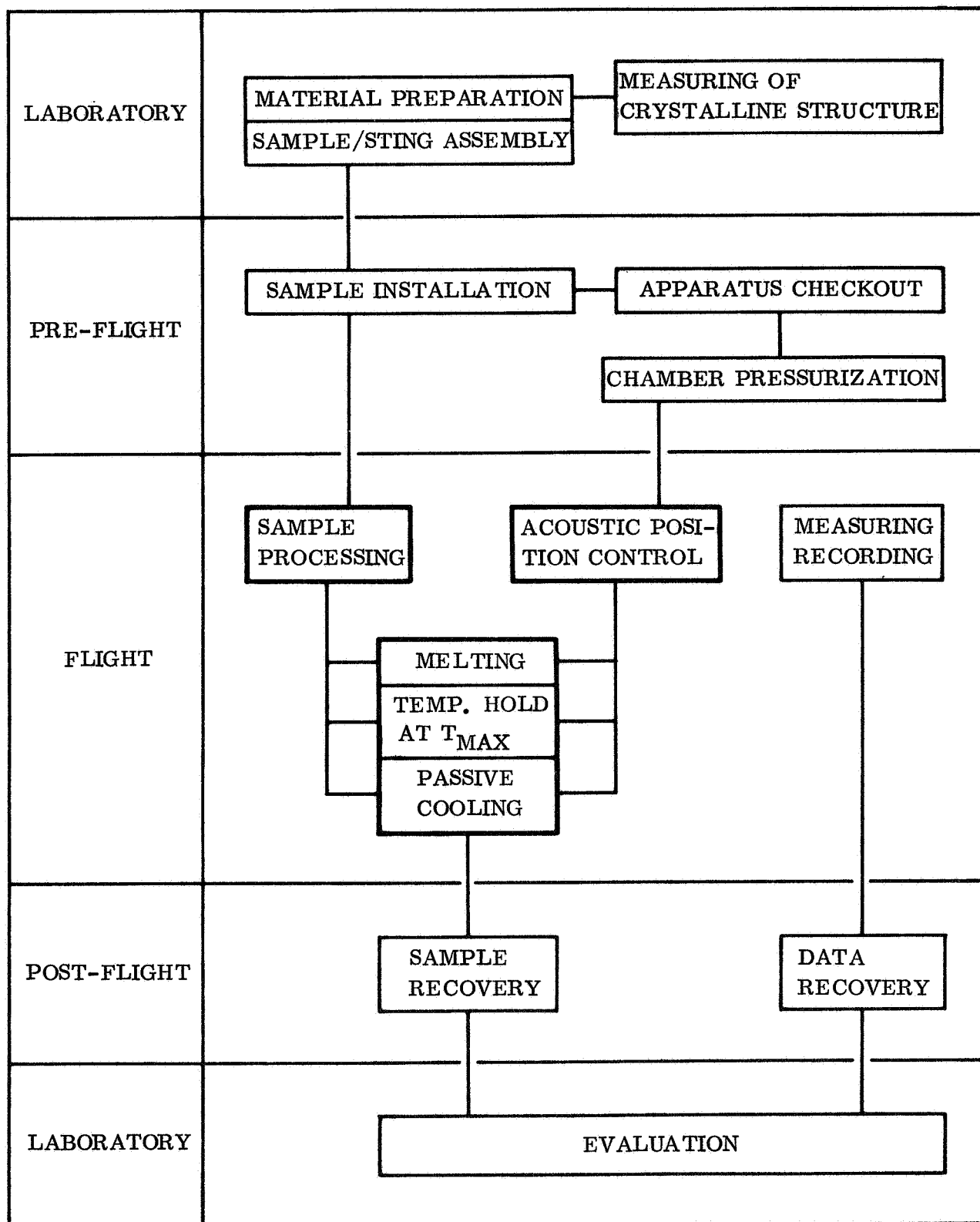


Figure 7.18-1. Process Flow Diagram - Chalcogenide Glasses



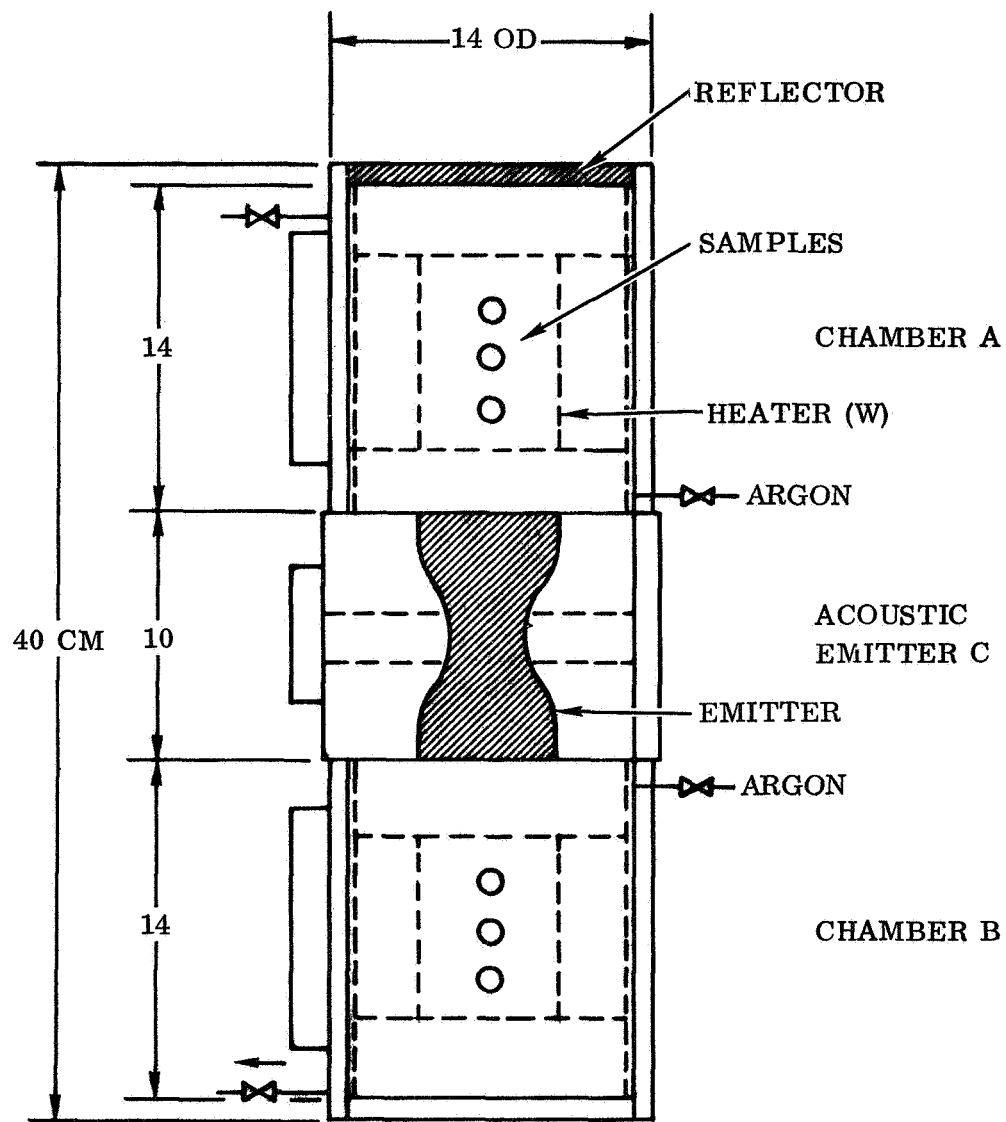


Figure 7.18-2. Processing Module for Experiments with Chalcogenic Glasses (Acoustic Position Control)

7.18-12

# ROCKET PAYLOAD ASSEMBLY

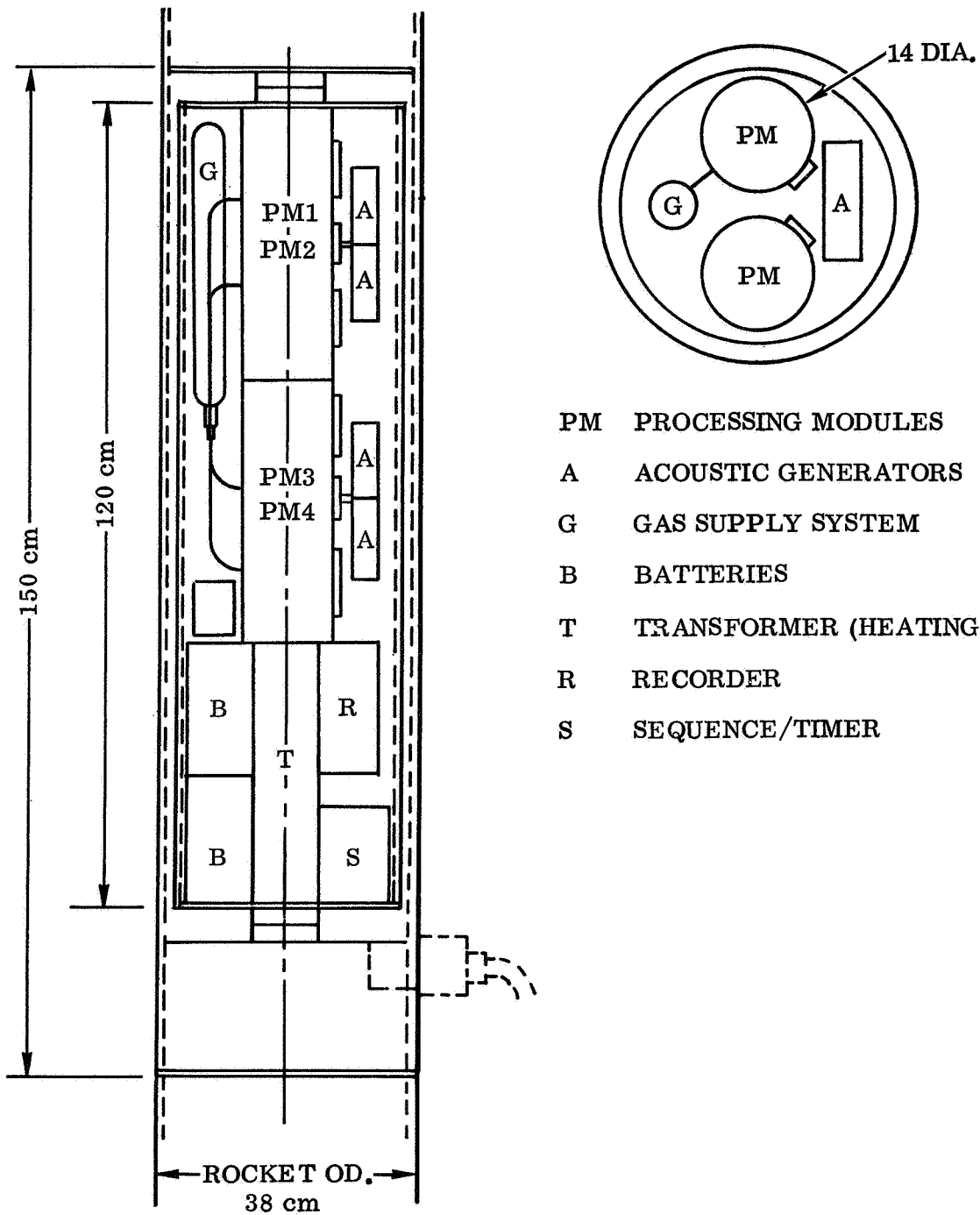


Figure 7.18-2. Dedicated Payload Assembly – Chalcogenic Glasses

# EXPERIMENT PROGRAM – TRAJECTORY B

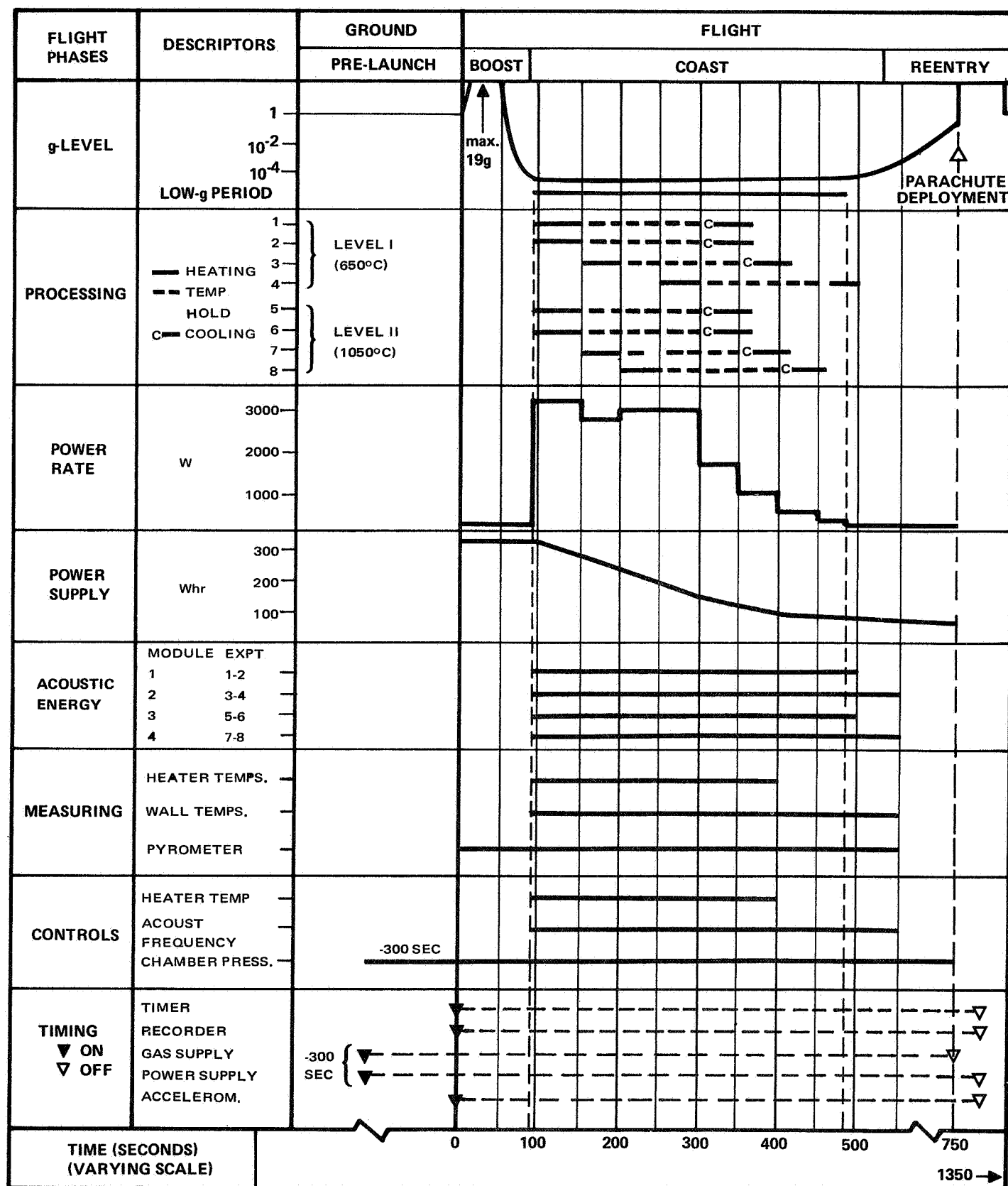


Figure 7.18-4. Chalcogenide Glasses

## 8. ROCKET EXPERIMENT PAYLOADS

The detailed evaluation of individual processes in Section 7 clearly indicates that most experiments call for minimum low-g times in the order of 40 to 360 seconds depending on the nature of the process. It was further found that these limited low-g forces are perfectly adequate for a conclusive process evaluation. With very few exceptions, longer low-g times merely permit an increase of the material quantity, not necessary for a first-order process evaluation. Rocket flights which provide the required minimum low-g time regime are, therefore, an effective tool for process evaluation and the definition of the property gains achievable by low-g processing.

Short low-g times in the order of several seconds, which can be obtained in drop towers and aircraft, are adequate only for exploratory experiments and the evaluation of individual process parameters. In only a few cases can complete processing be performed within a few seconds. Drop towers have been found preferable to aircraft in view of the reliable reproducibility of low-g conditions, not achievable in aircraft. Drop tower experiments have been identified for the following processes:

- Superconductors

- Metastable alloys - thermal dispersion

- Free alloying

- Free processing system

These experiments are, however, only of an exploratory nature since the necessarily small material quantity limits the accuracy of the evaluation measurements and, consequently, the conclusiveness of results.

The definition of experiment payloads in the following sections is, therefore, confined to rocket experiments or "extended low-g testing."

### 8.1 MAJOR PAYLOAD ELEMENTS

In Section 7, payloads have been defined for each experiment, predicated on the exclusive use of one rocket flight for the concerned process. Such "dedicated payloads" will, however, be a rare case. For highest cost effectiveness of a rocket test

program it will be desirable to integrate two or more different experiments into one payload and flight. The compatibility of various experiments for integration in a multi-purpose payload is determined by a limited number of essential experiment requirements and equipment characteristics, referred to as "payload elements." They represent the basic building blocks for the composition of dedicated and mixed payloads.

#### 8.1.1 Identification of Major Payload Elements

The process of payload definition, as it has been exercised in each experiment evaluation of Section 7, consists of two basic procedural instruments: (1) several sets of functional and physical data which represent the basic building blocks of the payload synthesis; they are designated as major payload elements, (2) the process and methods to integrate these elements into a specific payload within the constraints of operational and physical capabilities of specific research rockets and trajectories.

One of the prime objectives of this study was to define the major payload elements in numerical terms or sets of data. Once these basic building blocks have been established for each process, the definition of dedicated or mixed rocket payloads is merely a matter of integration by common method of systems engineering.

The purpose of this section is to identify the major payload elements. This is best achieved by a review of the payload definition procedures applied throughout Section 7 and illustrated in Fig. 8-1.

It may be well to first re-state the terminology for the major payload components, used in this report. The payload assembly consists of two major subassemblies: the apparatus and the support module. The standardized support module is the same for all types of apparatus, except for variations in the power supply and conditioning capabilities. The apparatus is the experiment subassembly and is composed of a number of individual processing modules. According to the composition of the apparatus, two types of payloads are distinguished.

- (1) A payload whose apparatus consists of identical process experiments is designated as dedicated payload. The concerned processing modules are always identical.

- (2) A payload whose apparatus comprises dissimilar process experiments is designated as mixed payload. In this case, the processing modules may be identical or mixed, depending on the experiment requirements.

The initial entries for the payload definition are two sets of data for a specific process, as they have been defined numerically for each experiment in Section 7. They are (1) the functional experiment requirements, such as low-g processing time or power input rate, and (2) the characteristics of the required processing equipment which, for the individual experiment, refer essentially to the processing module. They represent the first two major elements of the payload definition. They apply to dedicated as well as mixed payloads, except that in the case of mixed payloads two or more pairs of data sets are introduced.

These two payload elements provide the data for the computation of the apparatus characteristics. The apparatus definition requires further the determination of the number of identical (dedicated payloads) or dissimilar (mixed payloads) experiments that can be accommodated on one flight. The number of experiments in turn, is limited by the capabilities of the rocket and by the requirements and limitations of the support module. The applicable support module type and outfitting can usually be selected readily for the concerned experiments, since the only variation is the power supply and conditioning capability. Rocket capabilities and support module characteristics enter the evaluation as two additional sources of data or payload elements for the payload definition.

The number of experiments which determines apparatus and payload is then obtained by trade-off and iteration evaluations with the objective to achieve an optimum balance between experiment requirements, rocket capabilities and support module characteristics. All necessary data for this evaluation and, ultimately, the payload definition are provided by the four defined payload elements:

- (1) Functional experiment data
- (2) Processing module characteristics
- (3) Support module characteristics
- (4) Rocket payload capabilities.

In the evaluation, the requirements and capabilities are essentially represented by five criteria. The following table identifies these five trade-off criteria and the applicable payload elements which serve as data sources. It further indicates whether the criterion enters the trade-off as a requirement ("needed") or a capability ("available").

<u>Criteria</u>	<u>Expt. Rqmt's.</u>	<u>Processing Modules</u>	<u>Support Modules</u>	<u>Rocket Capabilities</u>
Total Low-g Time	Rqmt.			Cap'y.
Weight		Rqmt.	Rqmt.	Cap'y.
Space (P/L Height)		Rqmt.	Rqmt.	Cap'y.
Max Power Rate	Rqmt.		Cap'y.	
Total Power Consumpt.	Rqmt.		Cap'y.	

Once the number of experiments has been established, the definition of the operational and physical data of the multiple-experiment apparatus and the payload is merely a matter of computation and equipment arrangement. For mixed payloads, the procedure is essentially the same, except that we start with two or three different basic entries as to functional experiment requirements and module characteristics.

These five criteria listed above serve also as primary descriptors for the numerical definition of the payload. The additional descriptor of the payload definition is the number of samples per experiment and per flight. In many cases this differs from the number of experiments, since each processing module can accommodate more than one sample. For each processing module the samples may differ in material characteristics, but are subjected to identical processing conditions.

### 8.1.2 Designation of Payload Elements

To enhance the conciseness and manageability of the subsequent evaluation of various payload choices and to minimize repetitious explanations, it was found necessary to introduce abbreviations and symbols for the identification of functional and physical

payload elements:

- (1) Experiment Data, applying to the individual experiment (module) or complete payload, as specified in each case to min. low-g processing time for the defined material(s) and material quantity(ies), always expressed in seconds.

T <sub>max</sub>	Max. processing temperature (° C)
W	Max. energy rate (watts)
Wh	Total energy consumption (watt-hours)
SA/Expt.	Number of samples per experiment
SA/PL	Number of samples per payload

- (2) Apparatus Data applying - as specified - to individual modules, multi-module apparatus assemblies or the entire payload (P/L).

h	Payload space requirements, measured in axial height in the payload section (cm)
Wt	Weight in kg

- (3) Modules (for detailed description refer to section listed in parenthesis).

EPS	Stationary electrophoresis module (7.1.8)
EMP	Electro-magneto-phoresis module (7.2.8)
EF-1	Electrical resistance furnace 1 (6.2.1.1)
EF-2	Electrical resistance furnace 2 (6.2.1.2)
- - A	EF-1 or EF-2 with attachments (7.4.8, 7.6.8, 7.11.8)
EF-AG	Electrical resistance furnace with acoustic positioning (7.18.8)
EXO	Exothermic furnace (6.2.2)
DR	Direct-resistance furnace (6.2.3)
DRM	Direct resistance furnace modified for radiation heating (7.12.8)
DR-DRM	Dual DR furnace for combined radiation and direct-resistance heating (7.17.8)
FPS	Free processing system (7.15.8)
MEM	Membrane drawing module (7.16.8)



SPEC Special (single-purpose) modules

S/M Support module (5.0)

- (4) Power Conditioning Requirements (Support Module), identified by varying combinations of the following symbols:

B	Battery	(for data
I	Inverter	see table
T	Transformer	5-1)
R	Rectifier	

- (5) Rockets and Trajectories as defined in Section 4.2 and Table 4-1.

RR Research Rockets

RR-1 Aerobee 170

RR-4 Aerobee 200

Tr'y Trajectories

TR-A Min standard WSMR ( $t_o = 243$  sec)

TR-B Max standard WSMR ( $t_o = 390$  sec)

Experiments/Processes are identified by numbers 1 to 18 as introduced in Table 1 (Sect. 1), and as used as second digits in Sect. 7 (7.1 to 7.18).

Verification levels which represent significantly different requirements are identified by the addition of Roman numerals (such as "Free Alloying II or "14-II").

## 8.2 PAYLOAD ELEMENT DATA

This section is designed to furnish all necessary data for the selection and definition of rocket payloads and for the involved trade-off evaluations described in Sect. 8.1 1. It represents essentially an extraction and compilation of the applicable data defined in Sections 4 through 7 of this report.

For convenient retrieval, data are presented in chart form with a minimum of descriptive text. To enhance visibility, extensive use is made of the abbreviations introduced in Sect. 8.1.2.

### 8.2.1 Functional Experiment Requirements

The functional data for the experiments defined in Section 7 are summarized in Table 8-1. The data apply to the single experiment (one module), considered as building blocks for the composition of multiple-experiment payloads. They are confined to payload interface characteristics, as they evolved from the material and process evaluations of Section 7. All material- and process-related data are omitted since they are of no significance for the apparatus and payload composition.

The primary data for the computation of multiple payloads are the time and power requirements. It should, however, be remembered that these data are not necessarily additive, since the totals for a multiple payload depend on the experiment programming over the low-g and flight time. The achievement of minimum totals by optimized programming has been demonstrated throughout Section 7. The only data which are additive are the power consumption (wh) and the number of samples. As noted in the chart, the power data do not include support equipment (timer, recorder etc.) which is in the order of 100 to 150W, nor ground supplied power.

The g-level ("max. g") is simply a limit value. For mixed payloads, the g-level is dictated by the experiment with the lowest value.

### 8.2.2 Processing Modules

In order to arrive at meaningful experiment specifications, it was necessary to define and design in the process evaluations of Sect. 7 the processing device for each experiment in some detail. The design studies were aimed at (1) multiple use of devices for several experiments/processes, (2) full adaptation to low-g conditions, particularly with regard to the management of cooling fluids and (3) modular design in line with the principles outlined in Sect. 3. It was found that the same basic processing module can be used for a number of experiments and processes with minor modifications or attachments; in other cases the experiment requirement could only be met with a single-purpose module.

The resulting inventory of processing modules is summarized in Table 8-2. The table lists modifications, data, applicable experiments and the related support module type. Each module is, for the purpose of apparatus and payload synthesis, adequately defined by three interface characteristics:

Weight

Axial height, representative of module dimensions

Max. power rating

According to the basic apparatus design concept, all modules are stacked along the rocket/payload axis. The axial height is, therefore, the only required dimensional value. Only in two cases, the membrane drawing module (MEM-Expt. 16) and the furnace with acoustic position control (EF-AC, Expt. 18), the height comprises two modules side-by-side.

The final item of Table 8-2, a central argon supply system, has no dimensional requirements since it can, in all cases, be absorbed by apparatus cavities or other space reserves. Minor items, such as the steam exhaust system for exothermic furnaces, are not listed as their weight is included in the module weight.

### 8.2.3 Support Modules

The primary payload interface characteristics of the support module are weight and space requirements and power capabilities. The weight and space (height) requirements define the weight limitations and the available space for the apparatus. They are exclusively determined by the power supply and conditioning system as they are required for the experiments of a specific apparatus assembly. All other components of the support module are standard equipment which remains unchanged for all experiments.

The accurate definition of the support module data is part of the trade-off process discussed in 8.1.1. For a given set of experiments and apparatus assembly, the data are computed from Table 5-1 (Section 4.0), which identifies the weight and dimensions of each support module component. Typical support module data, as they evolved from the experiment evaluations in Section 7 are listed in Table 8-3. The correlation with

applicable processing modules in the last column of Table 8-3 should facilitate the selection of the support module for a planned payload, subject to revision during the payload optimization process.

#### 8.2.4 Rocket Capabilities

The rocket data required for payload synthesis are presented in Table 4-1 (Sect. 4.2). In the payload assessments of Section 7, preference was given to rocket classes 1 and 4 (Aerobee 170 and 200) for the following reasons: (1) They meet all initial experiment requirements, (2) long-time service and, consequently, high reliability, (3) extensive NASA experience and established launch facilities at White Sands Proving Ground.

The dimensional characteristics of the payload section are essentially identical for both types. Possible extension of the axial height or even diameter of the payload section was not considered in order to maintain a firm reference base. For the same reason only one trajectory was introduced for each rocket type. By selection of the minimum White Sands Missile Range trajectory for the Aerobee 170 and the maximum WSMR for the Aerobee 200, approximately equal payload weight capability was achieved. In this way the difference between the two rockets was concentrated on the most significant characteristic, the low-g time, specifying a minimum and maximum capability. Numerical data for the discussed rocket capabilities are listed below.

<u>Rocket Class</u> <u>Trajectory</u>	<u>1 (Aerobee 170)</u> <u>A (Min WSMR)</u>	<u>4 (Aerobee 200)</u> <u>(Max. WSMR)</u>
Payload Section O. D.	38 cm	38 cm
Payload Section Height	150 cm	150 cm
Payload Weight	130 kg	125 kg
Low-g Time	243 sec	390 sec

### 8.3 DEDICATED PAYLOADS

A payload consisting of a maximum number of identical experiments is designated as a dedicated payload. The payload assembly is composed of an apparatus with identical processing modules and the applicable support module type.

In the experiment evaluations of Section 7, dedicated payloads were defined in detail for each process and experiment. A summary of the most significant payload data is presented in Table 8-4. It identifies, for each experiment, the following characteristics:

<u>Operational Data</u>	Rocket class
	Trajectory
<u>Physical Data</u>	Processing module - type and number
	Support module type
	Payload weight
<u>Experiment Data</u>	Total low-g time
	Max. processing temperature
	Number of samples per flight

### 8.4 MIXED PAYLOADS

For highest effectiveness of a rocket test program it will be desirable to evaluate a wide variety of processes and materials in a minimum number of rocket flights. This places emphasis on mixed payloads comprised of two or more dissimilar experiments or sets of experiments. The objective of this section is to identify feasible and optimum experiment combinations, based on their physical, functional and operational compatibility. It serves as the basis for the program definition of Section 9, and, together with the payload element data of Sect. 8.2, for NASA programming activities.

#### 8.4.1 Experiment Compatibility

The compatibility of experiments or the adaptability to their combination in an apparatus of dissimilar experiments is determined: Primarily by the required power conditioning and, consequently, by the acceptability of a common support module; secondarily by physical and operational characteristics. Physical characteristics refer to the configuration and dimensions of the concerned processing modules. Operational characteristics refer to possible interference between experiments, such as vibrations introduced by an acoustic system.

The compatibility of experiments and the pertinent processing modules are identified in the right-hand section of Table 8-5. Interchangeable experiments and modules which can be readily combined into one apparatus are indicated by vertical connection lines. The chart indicates that only one experiment, chalcogenic glasses, is not compatible with any other experiment; it postulates a dedicated payload.

In the use of the chart it should be remembered that the support module data for a given set of experiments is independent of the number of experiments. For example, the total power consumption of the support module ("wh") is constant, whereas for the apparatus it is the sum of the individual experiment requirements.

The compatible experiments identified in Table 8-5 are arranged in groups in Table 5-6 for better visibility. Each group comprises the experiments which can be combined into mixed payloads.

It is apparent that there is a wide variety of feasible mixed payloads. The numerical definition of all possible payloads far exceeds the scope of this study. Numerical data are, however, specified for the mixed payloads of the proposed initial flight program discussed in Section 9.

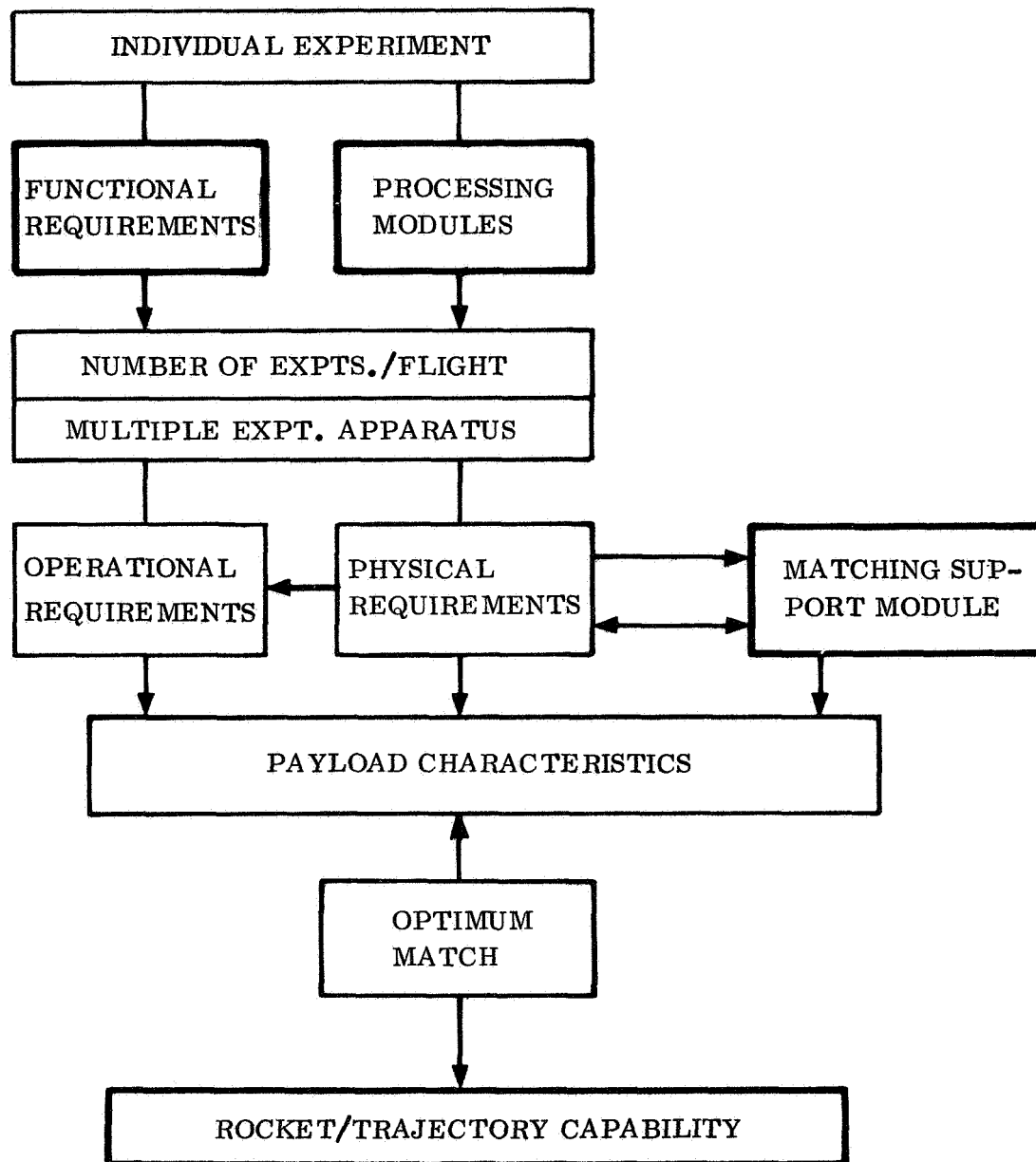


Figure 8-1. Major Elements of Payload Synthesis and Definition

Table 8-1. Individual Experiment Requirements (Single Experiment Module - Excluding Support Systems Requirements)

No.	Experiments	Time (sec)		Temp. Max. °C	Max. g	Power Rqts. <sup>2)</sup>		Samples Per Expt.	Process Module
		Low-g	Total			Max. W	Wh		
1	Bio-Electrophor. I	360	360	20	10 <sup>-4</sup>	340	35	4	EPS
	Bio-Electrophor. II	600	600	20	10 <sup>-5</sup>	340	57	4	EPS
2	Bio-Electro. Magn. Phor.	360	360	20	10 <sup>-5</sup>	400	59	1	EMP
3	Fiber/Part. Compos - Predisp.	130	1000 <sup>1)</sup>	700	10 <sup>-4</sup>	800	33	1-2	EF-2
	Fiber/Part. Compos. - Exothermic	130	150	1200	10 <sup>-4</sup>	100	5	1-2	EXO
4	Fiber/Part. Compos. - Low-g Mixing	100	1000 <sup>1)</sup>	700	10 <sup>-4</sup>	600	43	1	EF-2-A
5	Controlled Density Mat. - Predisp.	140	800 <sup>1)</sup>	700	10 <sup>-5</sup>	1600	47	1-2	EF-2
6	Controlled Density Mat. - Dynamic Foaming	150	850 <sup>1)</sup>	700	10 <sup>-5</sup>	1000	49	1	EF-2-A
7	Unidirectional Eutectics	390	550	650	10 <sup>-5</sup>	250	30	1	DRM
8	Superconductors	40	40	2200	10 <sup>-5</sup>	700	6	1	DR
9	Metastable All. - Thermal Disp.	240	1500 <sup>1)</sup>	1000	10 <sup>-4</sup>	650	35	1-2	EF-1
10	Metastable All. - Therm. Disp. - Hi Temp.	80	80	2500	10 <sup>-4</sup>	750	21	1	DR
11	Metastable All. - Homogenization	360	1500 <sup>1)</sup>	1000	10 <sup>-4</sup>	650	42	1	EF-1
12	Single Crystal Growth I	390	900 <sup>1)</sup>	550	10 <sup>-5</sup>	150	22	1	DRM
	Single Crystal Growth II	390	600 <sup>1)</sup>	1150	10 <sup>-5</sup>	400	47	1	DRM
13	Nucleation Research	390	600 <sup>1)</sup>	1150	10 <sup>-5</sup>	400	47	1	DRM
14	Free Alloying I	40	40	2500	10 <sup>-4</sup>	700	6	1	DR
	Free Alloying II	90	120	2500	10 <sup>-3</sup>	1800	160	1	FPS
15	Free Processing System	90	120	2500	10 <sup>-3</sup>	1800	160	1	FPS
16	Shaping/Membranes	100	900 <sup>1)</sup>	1100	10 <sup>-5</sup>	200	9	1	MEM
17	Oxide Glasses	50	80	2500	10 <sup>-5</sup>	1200	11	1	DR/DRM
18	Chalcogenide Glasses	240	240	1150	10 <sup>-3</sup>	1060	42	1-3	EF-AC

1) Including ground preheating and/or post-low-g cooling.

2) Flight (battery supplied) only.



Table 8-2. Processing Equipment Inventory

Processing Modules	Attach- ments	Max. Power Rat'g (W)	Wt (kg)	Ht (cm)	Sampl. Per Module	Support Mod. 1)	Applicable Experiments
Stationary Electrophoresis	—	340	24	35	4	7	1
Electro-Magneto-Phoresis	—	400	38	55	1	7	2
Electr. Furnace 1	—	650	14	18	2	4	9
	Mixg.	650	15	22	2	4	11
Electr. Furnace 2	—	1,600	13.5	16	1-2	4,5	3,5
	Mixg.	1,000	15	20	1-2	4,5	4,6
El. Furn. - Acoustic Position'g	—	1,100	9.5	40	2-6	6	18
Direct Resistance Furnaces	—	700	12	14	1	2	8, 10, 14
	Rad.	400	12.5	14	1	2	7, 12, 13
Dual Direct Res. Furnace	Rad.	950	14	14	1	2	17
Exothermic Furnace	—	50	13	18	1	1	3
Free Process'g System	—	1,800	15.5	18	1	5	14, 15
Membrane Drawing	—	800	10	10	1	1	16
Argon Supply System	—	—	6	— <sup>2)</sup>	—	—	All except 1 10 6

1) Support module type — Numbers refer to Table 8-3.

2) Space for argon supply system provided by payload cavities.

Table 8-3. Characteristics of Typical Support Modules

No.	Type	No. of Batter.	Stored Power (Wh)	Max. Power Rate (W)	Power Type to Apparatus	Weight	Weight	Applicable Processing Modules
1	B	1	110	1550	DC-28V	45	55	EXO, MEM
2	BI	1	110	1550	AC-1000 Hz	48	60	DR, DRM, DR/DRM
3	BI	2	220	3100	AC-1000 Hz	53	70	DR, DRM, DR/DRM
4	BIT	1	110	1550	AC	52	60	All EF <sup>1)</sup> , FPS
5	BIT	2	220	3100	AC	57	65	All EF <sup>1)</sup> , FPS
6	BIT	3	330	4000 <sup>2)</sup>	AC	60	70	All EF <sup>1)</sup> , FPS
7	BITR	1	110	1550	DC-1200V	53	60	EPS, EMP

1) EF-1, EF-2 and EF-AC

2) Limited by transformer size

TABLE 0-4. DEMONSTRATION FACILITIES

No.	Experiments	RR/Tr'y			$t_o$ (sec)	Max. Temp. (°C)	P/L Weight (kg)	Processing Modules		Sample per P/L
		1 A	4 B	C				Type	No.	
1	Stationary Electrophoresis I				360	-	100.5	EPS	2	8
	Stationary Electrophoresis II				600	-	103.5	EPS	2	8
2	Continuous Electrophoresis (Emp)				360	-	90.5	EMP	1	1
3	Fib/Part. Composites - Predisp.				240	700	124.5	EF-2	5	5 (10)
	Fib/Part. Composites - Exotherm.				240	1200	110	EXO	5	5 (10)
4	Fib/Part. Composites - Low-g mixing				240	700	119	EF-2-A	4	4 (8)
5	Contr. Dens. Met. - Predisp.				240	700	97.5	EF-2	3	3 (6)
6	Contr. Dens. Met. - Dynamic foaming				240	700	115	EF-2-A	4	4
7	Unidirectional Eutectics				390	1200	122.5	DR-MOD	5	5
8	Superconductors				240	2400	127	DR	6	6
9	Metastable Alloys - Th. D. - Low Temp				390	1000	117	EF-1	4	8
10	Metastable Alloys-Therm. Disp-Hitemp.				240	2400	130	DR	6	6
11	Metastable Alloys - Homogenization				390	800	119	EF-1-A	3	6
12	Single Crystal Growth I				390	600	119.5	DR-M	5	5
	Single Crystal Growth II				390	1200	122.5	DR-M	5	5
13	Nucleation/Research				390	1200	122.5	DR-M	5	5
14	Free Alloying I				240	2400	127	DR	6	6
	Free Alloying II				360	2400	122	FPS	4	4
15	Free Processing System				360	2400	122	FPS	4	4
16	Shaping/Forming				240	800	108	Spec	6	6
17	Oxide Glasses				390	2500		DR-DRM	5	5
18	Chalcogenide Glasses				390	1150	108	EF-AC	4	8-24

Experiments		Expt. Module Data				Compatibility x)						Sample per Expt.
		Type	h cm	Wt Kg	Wh	I	II	III	IV	V	VI	
1	Stat. Electrophoresis	EPS	35	22	35	●						4
2	Electro-Magneto - Phoresis	EMP	55	38	59	●						1
3	Composites - Predispersed	EF-2	16	13.5	33/45		●					1-2
	Composites - Predispersed	EXO	18	13	1					●		1-2
4	Composites - Low-g mixing	EF-2-A	20	15	43		●					1
5	Contr. Dens. Met's. - Predispersed	EF-2	16	13.5	47		●					1-2
6	Contr. Dens. Met's. - Dynamic foaming	EF-2-A	20	14.5	49		●					1
7	Unidirectional Eutectics	DRM	14	12.5	47			●				1
8	Superconductors	DR	14	12	6			●				1
9	Metast. Alloys - Thermal dispersn.	EF-1	18	14	35		●					1-2
10	Metast. Alloys - Therm. D. - Hitemp.	DR	14	12	21			●				1
11	Metast. Alloys - Homogenization	EF-1-A	20	15	42		●					1
12	Single Crystal Growth	DRM	14	12.5	22/47			●				1
13	Homogeneous Nucleation	DRM	14	12.5	43			●				1
14	Semi-Free Alloying	DR	14	12	6			●				1
	Free Alloying	FPS	18	15.5	40				●			1
15	Free Suspension System	FPS	18	15.5	40				●			1
16	Shaping - Membranes	MEM	25	10	10					●		1
17	Oxide Glasses	DR-DRM	14	14	11			●				1
18	Chalcogenide Glasses	EF-AC	40	9.5	30/76						●	2-6
Support Module Data		Type	Wh			25	25	25/50	38	25	25	
			Wt - Kg			53	57	48	57	45	60	
			Ht - cm			60	70	60	70	55	70	
						BITR	BIT	BI	BIT	B	BIT	

x) Roman numerals refer to groups of Table 8-6

Table 8-6. Groups of Compatible Experiments for Mixed Payloads

Experiment/Process Groups	Processing Modules	Support Module
<u>Group I: Electrophoresis</u>		
1 Stationary Electrophoresis	EPS	BITR
2 Electro-Magneto-Phoresis	EMP	
<u>Group II: Electric Furnaces/Active Cooling</u>		
3 Composites — Predispersed	EF-2	BIT
4 Composites — Low-g Mixing	EF-2-A	
5 Controlled Dens. Metals — Predispersed	EF-2	
6 Contr. Dens. Metals — Dynamic Mixing	EF-2-A	
9 Metastable Alloys — Therm. Dispers.	EF-1	
11 Metastable Alloys — Homogenization	EF-1-A	
<u>Group III: Direct Resistance Furnaces/Passive Cooling</u>		
7 Unidirectional Eutectics	DRM	BI
8 Superconductors	DR	
10 Metastable Alloys — Therm. Disp. - Hi-Temp.	DR	
12 Single Crystal Growth	DRM	
13 Homogeneous Nucleation	DRM	
14 Semi-Free Alloying	DR	
17 Oxide Glasses	DR-DRM	
<u>Group IV: Free Processing System</u>		
14 Free Alloying	FPS	BIT
15 FPS Equipment Development	FPS	
<u>Group V: Unrelated Experiments</u>		
3 Composites — Exothermic Heating	EXO	B
16 Shaping — Drawing of Membranes	MEM	
<u>Group VI: Incompatible Experiment</u>		
18 Chalcogenide Glasses	EF-AC	BIT

## 9. ROCKET TEST PROGRAMS

The primary objective of a rocket test program is twofold: First, to evaluate the feasibility and/or effectiveness of a wide variety of candidate processes; since this is accomplished by experimental processing under true low-g conditions, the results should permit a reliable judgement as to the practical promise of each process and to the relative value of processes

The second objective is to obtain data on g-sensitive process and materials parameters required for process improvement and optimization.

There is, however, a third objective whose importance should not be underrated: The generation of experience and data on equipment design and operation.

The combined results will provide a reliable basis for the final definition of shuttle-based experiments and for the design of the related space facilities.

The following sections discuss the programming criteria and procedures, which are then applied to the formulation of a typical Phase I program

### 9 1 PROGRAMMING CRITERIA

An effective program is characterized by an optimum balance between desirable objectives and certain constraints. The most significant desirable objectives are:

- (1) Maximum variety of processes at a minimum number of flights.
- (2) High number of experiments (processing conditions) and samples.
- (3) Processing facilities reproducing all essential process parameters
- (4) High functional reliability of the payload assembly and high assurance of experiment success.
- (5) Earliest integration of each process in the program and earliest availability of test results

The degree to which these objectives can be realized is determined by the necessary compliance with a number of constraints, such as:

- (1) Time of equipment availability

- (2) Minimum equipment inventory
- (3) Gradual capability build-up with regard to experiment and equipment sophistication and the related operational experience.
- (4) Physical compatibility of processing modules within one payload
- (5) Functional compatibility (potential interference) of experiments within one payload.
- (6) Availability of rockets (incl. refurbishment for re-use) and launch facilities.

## 9.2 EARLIEST EXPERIMENT READINESS

For all candidate processes discussed in Section 7, the state-of-art has been sufficiently advanced as a result of NASA-sponsored developmental programs, so that experiments could be prepared within a comparatively short time. In all cases the time required for the definition of specific materials, processing specifications and evaluation procedures, and for the preparation of samples is less than the time required for the preparation of the experiment apparatus. The time of experiment readiness is, therefore, solely determined by the earliest time of equipment availability, or the time required for:

Design of processing modules and apparatus

Fabrication of one prototype module

Module testing and checkout

Fabrication of additional modules

Apparatus assembly and check-out

The total time required for each type of apparatus varies with the state-of-art or the necessity for developmental efforts, and with the time required for each phase of preparation. With regard to the state-of-art, the equipment inventory may be divided into four-groups as follows (numbers in parenthesis identify processes):

- A. Functional characteristics and design are well established. Effort consists essentially in the detail design and fabrication of the apparatus.

Stationary Electrophoresis (1-I, 1-II)

Electro-Magneto-Phoresis (2)

- B. Design within state-of-art, however limited developmental effort is required.
  - Exothermic Furnace (3-II)
  - Direct Resistance Furnace DR (8, 10, 14-I)
  - Direct Resistance Furnace with Radiation Heating Attachment (7, 12, 13)
- C Conceptual Design established, however certain functional details require more extensive developmental effort (such as closed cooling system)
  - Electric Furnace EF-1 (9, 11)
  - Electric Furnace EF-2 (3, 4, 5, 6)
  - Dual Heating Furnace DR-DRM (17)
  - Membrane Drawing Apparatus (16)
- D. Considerable developmental effort required before design can be finalized.
  - Free Processing System (14-II, 15)
  - Electric Furnace with Acoustic Position Control (18)

Considering the required efforts to advance each device from the present state to an operational capability, the preparation time has been computed for each type of apparatus. The resulting times, which represent estimated experiment readiness, are presented in Fig. 9-1. The figure is arranged according to the previously established listing of processes for convenient correlation with Section 7 and the subsequent sections.

The figure shows, that earliest readiness is achieved for processes using the single-transformer direct-resistance furnaces (DR, DRM) and the exothermic furnace (7-9 months). This first availability range further includes stationary electrophoresis experiments, whose early readiness is due to existing apparatus designs and high functional predictability established in privately sponsored efforts (GDCA-UCSD).

The second group, 10-12 months, comprises various processes using electrical radiation furnaces (EF-1, EF-2), oxide glasses using the dual-direct-resistance furnace (DR-DRM) and continuous electrophoresis by the electro-magneto-phoresis technique.

The only processes which require more than one year of preparation time are chalcogenide glasses (acoustic positioning), experiments with the free processing system and free-alloying experiments using that system.



While these time definitions may appear rather optimistic, it should be remembered that early experiment performance was one of the prime considerations in all phases of this study. It was introduced as a constraint in the selection and sophistication of experiments, in the selection of materials and processing conditions and in the apparatus definitions.

Two qualifications may further be in order. (1) all time definitions use the so-called work-go-ahead as starting point; they exclude the unpredictable time requirements for appropriation of funds, RFQ's and contract negotiations. (2) In all time assessments an adequate funding of efforts was presumed.

### 9.3 PROGRAM DEVELOPMENT

The "payload elements" discussed in Section 8 provide the data for the technical definition of payloads; the lead-time requirements outlined above serve as primary guide for the sequencing of these payloads into a flight test program.

The primary data required for the definition and computation of payloads and for sequencing of these payloads into a flight program are listed below. The table identifies the characteristics which enter numerical trade-offs and computations (C = capabilities, R = Requirements), as well as the tables of Section 8 which serve as data sources. For convenience, the most frequently needed equipment data are compiled in Fig. 9-2.

		(Table)
<u>Rocket Capabilities</u>		
Low-g time	C	(4-1)
Payload height limitation	C	(4-1)
Payload weight limitation	C	(4-1)
<u>(Single) Experiment Data</u>		
Low-g time	R	( 8-1)
Maximum power rate		(8-1)
Board-power consumption		(8-1)
Maximum g-level		(8-1)
Applicable processing module(s)		(8-1)

(Table)

<u>Processing Modules</u>		
(Axial) height	R	(8-2)
Weight	R	(8-2)
Number of samples		(8-2)
Applicable support module		(8-2)
Compatibility with other modules		(8-5)
Earliest availability		(9-1)
<u>Support Modules</u>		
(Axial) height	R	(8-3)
Weight	R	(8-3)
Maximum discharge rate		C (8-3)
Total stored power		C

The definition of payloads, which has been discussed in detail in Section 8, consists primarily in the determination of the type and number of experiments and related processing modules which can be accommodated in one flight. This is accomplished by trade-offs between the requirements of experiments and processing modules, and the capabilities of vehicles and support modules as to low-g time, height, weight and power. For dedicated payloads, the number of modules and experiments and all pertinent data have been defined for each process in Section 7 and summarized in Table 8-4. For mixed payloads, the determination of the type and number of experiments and modules further calls for the consideration of compatibility (Table 8-5). Once the type and number of experiments has been established, the yield of the flight as to the number of material samples can be defined which serves as a measure of flight effectiveness.

The arrangement of the so-conceived payloads into a sequence of flights is then primarily governed by the earliest availability of processing modules or "experiment readiness" defined in the subsequent section 9.2, and secondarily by judgement as to other programming criteria listed in Section 9.1. The secondary reliance on judgement by the programmer or a program committee is due to the nature of the involved criteria which do not lend themselves to numerical representation and are subject to opinion and policy fluctuations.

## 9.4 EXAMPLE OF AN INITIAL TEST PROGRAM

In the following sections, an initial flight test program is formulated which integrates all processes discussed in Section 7 with the sole exception of process 7 (unidirectional eutectics). It is the result of extensive programming studies and the evaluation of numerous program arrangements, from which the presented program emerged as the most effective choice. The authors are, however, aware that there may be other, equally effective program choices and that shift in the relative emphasis of processes or fluctuations in funding may dictate other choices. For this reason the presented choice should be regarded as a typical program designed for the sole purpose of demonstrating the effectiveness of rocket flights for the verification of space manufacturing processes.

### 9.4.1 Assumptions

Besides the programming criteria discussed before, the program definition was based on the following assumptions:

1. Procurement of 6 Aerobee-200 rockets. Of these, 5 are assigned to the initial program; assuming conservatively 2 flights per vehicle (one refurbishment) this permits the performance of 10 flights. The remaining rocket serves as a standby in case of vehicle difficulties which would result in severe program delays; otherwise it will - as any other re-usable vehicles - be used in the continuing program.
2. The "initial" program represents the first phase of a continuing program. The extended service and write-off of payload hardware justifies the establishment of a substantial equipment inventory during Phase I. This, in turn, increases the pay-off of Phase I by the generation of extensive equipment performance data, useful not only for continued rocket experiments, but also for the definition of orbital facilities.
3. The lead time of each experiment is solely determined by the earliest availability of the concerned processing module. All process developments are sufficiently advanced that flight samples can be delivered in time.

Likewise, the support module, already in the state of design studies, will be available at the time of the earliest experiments (7 months from program start).

#### 9.4.2 Phase I Program Formulation

In the design of a typical Phase I program it was attempted to (1) represent, if possible, all candidate processes within 10 flights and (2) to obtain the highest number of individual experiments and material samples.

The number of experiments assigned to each process was determined by the following considerations:

- (1) The significance of the process and its expected product.
- (2) The desirability of several processing conditions.
- (3) The number of samples obtainable in the individual experiment.
- (4) Equipment limitations (time of availability and number of processing modules).

The combination of experiments into individual payloads was primarily determined by the equipment compatibility, as defined in Sect. 8.4.1 and Table 8-5, and secondarily by functional compatibility or potential interference. The sequencing of these payloads or flight assignment was dictated by the earliest equipment availability.

The basic structure of the resulting 10-flight program is shown in Fig. 9-3. It identifies the correlation of each payload composition and payload placement, shown in the center portion of the figure, with the modules required for each process listed at left, and the earliest availability of these modules indicated in the top section. The primary reasons for the selected composition and timing of each payload are substantiated in the following discussion of each flight. For conciseness, the processes are identified by the previously assigned numbers.

Flight 1. The first available module is the direct resistance furnace (DR). The first payload comprises, therefore, the experiments using this module (processes 8, 10 and 14).

Flight 2. The next available module is the exothermic furnace (EXO). The rather uncontrollable heating profile (in this simplified version) is acceptable only for

process 3. Since this module is further not compatible with any other, Flight 1 represents a dedicated payload of process 3 experiments

Flight 3. In the meantime, the modified version (radiation attachment) of the direct resistance furnace (DRM) is available, accommodating processes 7, 12 and 13 in the same payload with those of Flight 1. Process 7 has been eliminated in view of the marginal adequacy of the available low-g time. Flight 3 emphasizes process 12 (single crystal growth) with 3 experiments, in addition to one experiment each of processes 8, 10 and 13.

Flight 4. The relatively early availability of the stationary electrophoresis module (EPS) permits the performance of process 1 experiments in Flight 4. Temperature sensitivity precludes combination with furnace modules. Flight 4 is, therefore, a dedicated payload of two process 1 experiments

Flight 5. By the tenth month the basic version of the more sophisticated electric radiation furnaces EF-1 and EF-2, accommodating processes 5 and 9, are ready for use. Since they are fully compatible, a mixed payload of process 5 and 9 experiments are scheduled for this flight.

Flight 6 is a repeat of Flight 3, providing more experiments on single crystal growth (12) and nucleation research (13). The payload, consisting of DR and DRM modules, also provides for one additional experiment each of processes 8 and 10.

Flight 7. The availability of the mixing and dynamic foaming attachments (A) for the electric furnaces EF-1 and EF-2 permits at this point the performance of experiments on processes 4, 6 and 11.

In view of the limited number of experiments possible in payloads with furnaces EF-1 and EF-2, only an average of 2 experiments have been scheduled so far for each of the concerned processes 3, 4, 5, 6, 9 and 11. It may appear desirable to include one more payload of this type in the 10-flight program. It was, however, considered advisable to utilize the remaining three flights for processes which have not yet been included in the program in view of the long equipment lead times, and to rely on the continuing program (Phase II) for additional experiments on processes 3 to 6, 9 and 11

Flight 8 introduces experiments with oxide glasses (process 17), using the second modification of the basic DR module into a dual heating system (DR-DRM), which is compatible with other types of the DR-furnace family. The flight provides for 3 experiments on process 17 and one additional each on processes 12 and 13.

Flight 9. At the same time the electro-magneto-phoresis and the membrane drawing modules are ready for use. While they are compatible mechanically, there was some doubt as to the thermal compatibility. However a mixed payload of processes 2 and 16 experiments appeared permissible in view of the modest amount of heat involved in the MEM module and the complete absence of vibrations.

Flight 10. The final payload of the Phase I program combines the two long-lead-time modules, the electric furnace with acoustic position control (EF-AC) and the free processing system (FPS), comprising processes 15 and 18. The mixed payload is permissible with regard to interference, since the substantial difference in the frequency level (15-20 KHz and 300-600 KHz) is not expected to pose any problem.

The number of experiments per flight for each flight and each process are listed at the bottom and the right margin of Fig. 9-3. The complete 10-flight program comprises a total of 48 experiments. Also listed at the bottom of Fig. 9-3 is the gradual increase of tested processes as the program progresses.

#### 9.4.3 Program Plan and Data

A more detailed overview and data summary of the 10-flight program is presented in the "Program Master Plan," in Fig. 9-4. It consists of four major data blocks: The top section contains flight information, such as trajectory and min. low-g time. The second section identifies the payload equipment (support module type, processing modules, payload weight and axial height).

The most significant data are summarized in the third section. First, it conveys for each flight a picture of the apparatus assembly, each square representing one processing module. Each square contains two figures: the process number and the number of material samples (in parenthesis).

In the final (bottom) section of Fig. 9-4 the number of experiments and samples of each flight are broken down according to major material categories. It further identifies the total number of experiments and samples for each flight, each material category and the complete Phase I program.

As a rule, the number of experiments (processing conditions) is identical with the number of processing modules, i. e. one experiment per module. The only exception is process 18, whose processing module (EF-AC) has two chambers and accommodates two different experiments. (This accounts for the four experiments listed under Flight 10 - glasses).

Each "sample" represents one material composition. The number of samples per experiment varies with the nature of the process, the processing technique and the sample size required for evaluation measurements. For example, the direct-resistance heating technique (DR modules) limits the number of samples to one per module and experiment. In electric radiation furnaces (EF modules) up to three samples can be processed in the same experiment. The acceptable sample number depends then on the experiment objective. If the prime objectives are microstructural or electroactive properties, the sample can be small and more than one sample per experiment are possible, as in the case of processes 9 and 11 (metastable alloys). If, as in the case of some composite experiments, the evaluation of mechanical properties calls for a large sample size, only one sample can be accommodated in each module.

#### 9.4.4 Program Capabilities

The following table summarizes the program capabilities in terms of the number of processes, experiments and samples for each major material and process category. The classification of processes is somewhat more detailed than in Fig. 9-4 and is adapted to the commonly used identification of major areas of zero-g processing.

	<u>Processes</u>	<u>Experiments</u>	<u>Samples</u>
Electrophoretic separation of biological materials	2	3	9
Composites and controlled density metals (foams)	4	11	15
Superconductors	1	4	4
Metastable alloys (immiscibles)	3	8	11
Single crystal growth incl. kinetics of nucleation	2	10	10
Free processing of metals incl. systems testing	2	3	3
Free processing of glasses	2	7	11
Free forming	1	2	2
	<hr/>	<hr/>	<hr/>
Phase I Program	17	48	65

It was stated initially that the prime program objective is the representation of a wide variety of processes and processing conditions (experiments) in a limited number of flights. This is well achieved in the formulated program with 17 processes and 48 experiments in 10 flights. An equally valid measure of the program effectiveness is the number of samples, as they provide, by way of ground evaluation, the prime data source for the assessment of process capabilities and for the prediction of its pay-off in the form of products. The total program yield of 65 samples represents an average sample rate of 6.5 samples per flight.

A secondary pay-off of the program, which should, however, be not underrated, is the testing of processing equipment under low-g conditions. The program will produce data and experience on the following primary processing techniques.

(Techniques specially designed for low-g operations)

High-frequency position control and induction heating

Acoustic position control



Three active cooling techniques

Two low-g mixing techniques

Dynamic foaming

Liquid-state forming (membranes)

(Other significant techniques)

Two electrophoretic separation techniques

Four techniques of electric radiation heating

Direct-resistance heating

Dual (resistance and radiation) heating

Exothermic heating

## 9.5 PHASE I PROGRAM SCHEDULES

In the following schedules an independent time scale is used, either in terms of program months or sequence of flights. As in the program plan, it is assumed that all critical lead time efforts are initiated simultaneously.

### 9.5.1 Equipment Schedules

The 10-Flight Program calls for the following equipment inventory (listed in the order of first payload integration):

#### Basic Modules

- 2 Support modules with ground support, to be used alternately and refurbished between flights where indicated.
- 6 Direct-resistance furnaces (DR)
- 5 Exothermic furnaces with open cooling system
- 2 Stationary electrophoresis modules (EPS)
- 2 Electric radiation furnaces, type 1, with closed cooling system (EF-1)
- 3 Electric radiation furnaces, type 2, with closed cooling system (EF-2)
- 3 Dual resistance and radiation heating furnaces (DR-DRM)
- 1 Electro-magneto-phoresis module (EMP)

- 2 Membrane drawing modules (MEM)
- 2 Electric furnaces with acoustic position control system and 2 processing chambers (EF-AC)
- 2 Free processing modules (FPS)

#### Attachments and Modification Kits

- 4 Modification kits for DR modules, to convert to radiation heating (DRM)
- 2 Ultrasonic mixing attachments for electric furnaces EF-1 and EF-2
- 2 Foaming attachments for electric furnace EF-2

#### Interchangeable Support Module Components

- 3 Transformers of varied size and output
- 4 Solid-state inverters of varied size and output
- 1 High-voltage rectifier
- 16 Yardley silver cell battery packs (1 to 3 per flight). Battery packs can be split to fit into cavities.
- 2 Ground support connectors.

Since the program formulation was based on the earliest equipment availability, the time of first flight integration is identical to the earliest availability defined in Fig. 9-1. The schedule of flight integration and, if applicable, refurbishment and re-use of each module as identified in Fig. 9-5.

#### 9.5.2 Program Performance Schedule

A typical schedule for the 10-flight program in terms of program month is formulated in Fig. 9- 6. Each flight is divided into four periods:

- (1) Equipment preparation, comprising design, fabrication and check-out.
- (2) Payload assembly: support module modification, if necessary, apparatus installation and payload check-out.
- (3) Flight performance
- (4) Evaluation of samples and flight recordings, documentation of results.

The schedule covers a 2-year period, from the start of processing equipment development to the documentation of the last flight. The only exception is the support module whose preparation has to precede the program start by two months to allow adequate checkout and to meet the date of the first payload integration. This is acceptable since specifications can be defined early on the basis of design studies already in progress.

Launch and flight activities extend over a twelve month period, presumably to continue in the next program phase. The spacing of individual flights in one or two months intervals is determined by equipment refurbishment requirements. This represents a minimum spacing which is, of course, flexible and subject to range availability. The flight spacing could be further compressed - or the payload assembly and check-out periods extended - by the availability of a third support module.

According to this schedule, the first test results would be available one year after program start. The accumulation of test results over the ensuing 12-month period is illustrated in Fig. 9-7 in terms of the number of evaluated processes, experiments (processing conditions) and samples (material compositions).

Experiments		Processg. Module	Earliest Readiness (Months from work start)			WSMR Traject.	Sampl. Per Module	Max. Modules Per P/L
			7-9	10-12	Over			
	Stat. Electrophoresis I	EPS	9			B	4	2
	Stat. Electrophoresis II	EPS	9			C	4	2
1	Continuous Electrophor.	EMP		12		B	1	1
1	Composites - Predispersed	EF-2		10		A	1-2	5
	Composites - Exotherm. Heatg.	EXO	8			A	1-2	5
	Composites - Low-g Mixing	EF-2-A		12		A	1-2	4
	Contr. Dens. Metals - Predisp.	EF-2		10		A	1-2	3
	Contr. Dens. Metals - Dynamic Foaming	EF-2-A		12		A	1	4
	Unidirectional Eutectics	DR-M	9			B	1	5
	Superconductors	DR	7			A	1	6
	Metastable All. - Therm. Disp.	EF-1		10		B	2	4
0	Metastable All. - High Temp.	DR	7			A	1	6
1	Metast. All. - Homogenization	EF-1-A		12		B	2	3
2	Single Crystal Growth	DR-M	9			B	1	5
3	Homogenous Nucleation	DR-M	9			B	1	5
4	Semi-Free Alloying	DR	7			A	1	6
	Free Alloying	FPS			15	B	1	4
5	Free Processing System	FPS			15	B	1	4
6	Drawing of Membranes	MEM		11		A	1	6
7	Oxide Glasses	DR-DRM		12		B	1	5
8	Chalcogenide Glasses	EF-AC			14	B	2-6	4

**Figure 9-1. Earliest Readiness of Experiments (Based on Earliest Equipment Availability)**

Basic Modules	Modifications (Attachm.)	Code	Ht (cm)	Wt (kg)	Earliest Availab. <sup>2)</sup> (mo)	Applic. Expts. No.	Support Mod.			Qt'y of Samp.
							Type 3)	Ht cm	Wt Kg	
Stat. Electrophoresis	—	EPS	35	24	8	1	7	60	53	4
Electro-Magn. -Phores.	—	EMP	55	38	12	2	7	60	53	1
Electr. Furnace EF-1	—	EF-1	18	14	10	9	5	65	57	2
	Mixing	EF-1-A	22	15	10	11	5	65	57	2
Electr. Furnace EF-2	—	EF-2	16	13.5	10	3,5	5	65	57	1-2
	Mixing	EF-2-A	20	15	10	4	5	65	57	1-2
	Foaming	EF-2-A	20	14.5	12	6	5	65	57	1-2
EF-Acoustic P.C. 1)	—	EF-AC	40	9.5	15	18	6	70	60	2-6
Direct Resistance Furnaces	—	DR	14	12	7	8, 10, 14	2	60	48	1
	Rad. Heatg.	DR-M	14	12.5	9	7, 12, 13	2	60	48	1
	Dual Heatg.	DR-DRM	14	14	12	17	2	60	48	1
Exothermic Furnace	—	EXO	18	13	6	3	1	55	45	1-2
Free Processing Syst.	—	FPS	18	15.5	15	14, 15	5	65	57	1
Membrane Drawing 1)	—	MEM	25	10	12	16	1	55	45	1

1) Same height accommodates 2 modules side-by-side

2) See text for qualifications

3) Numerals refer to Table 8-3

Figure 9-2. Equipment Data For Program Planning

Flight	1	2	3	4	5	6	7	8	9	10	1-10
Payload (Dedicated/Mixed)	Mix	Ded	Mix	Ded	Mix	Mix	Mix	Mix	Mix	Mix	Number of Expts.
Longest Lead Time Module	DR	EXO	DRM	EPS	EF	DRM	EF-A	DR/DR	EMP	FPS	
Earliest Availability	7	7	9	9	10	9	12	12	12	15	
1 Stat. Electrophoresis				(2)							2
2 Contin. Electrophoresis									(1)		1
3 Composites - Predispers. I											5
4 Composites - Predispers. II		(5)					(1)				1
5 Composites - Low-g Mixing											3
6 Contr. Dens. Met. - Predisp.					(3)		(2)				2
7 Contr. Dens. M. - Low-g Foaming											
8 Unidirectional Eutectics											4
9 Superconductors	(2)		(1)			(1)					2
10 Metastable All. - Therm. Disp.	(3)		(1)		(2)						5
11 Metast. All. - Hi-Temp. Disp.							(1)				1
12 Metast. All. - Homogenization								(1)			6
13 Single Crystal Growth			(3)			(2)		(1)			4
14 Homogeneous Nucleation			(1)			(2)					1
15 Free Alloying - I	(1)										2
16 Free Alloying - II											2
17 Free Processing System									(2)		3
18 Free Forming + Membranes											4
19 Oxide Glasses											
20 Chalcogenide Glasses											
Number of Experiments	6	5	6	2	5	6	4	5	3	6	48
Represented Processes (Cumulative)	3	4	6	7	9	9	12	13	15	17	

Note: Numbers in circles represent number of experiments.

Figure 9-3. Program Structure



FLIGHT/PAYLOAD EQUIPMENT MODULES	1	2	3	4	5	6	7	8	9	10
PROGRAM START										
2 SUPPORT MODULES	SM --	SM --	SM --	SM --	SM --	SM --	SM --	SM --	SM --	SM --
6 DIRECT RESISTANCE FURNACES	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
3 DUAL HEATING FURNACES	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --	DR --
5 EXOTHERMIC FURNACES	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --
	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --
	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --
	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --	EXO --
2 ELECTRIC RADIATION FURNACES TYPE 1	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --	EF-1 --
3 ELECTRIC RADIATION FURNACES TYPE 2	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --
	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --	EF-2 --
3 ELECTROPHORESIS MODULES	EPS --	EPS --	EPS --	EPS --	EPS --	EPS --	EPS --	EPS --	EPS --	EPS --
2 MEMBRANE DRAWING MODULES	MEM --	MEM --	MEM --	MEM --	MEM --	MEM --	MEM --	MEM --	MEM --	MEM --
2 ELECTRIC FURNACES WITH ACOUSTIC POSITION CONTR	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --	EF-AC --
2 FREE PROCESSING SYSTEMS	FPS --	FPS --	FPS --	FPS --	FPS --	FPS --	FPS --	FPS --	FPS --	FPS --

CODE:  
 DESIGN, FABRICATION  
 AND CHECK-OUT  
 -- -- --  
 RE-USE  
 (UNCHANGED) --  
 REFURBISHMENT  
 --R--  
 MODIFICATION  
 --M--

Figure 9-5. Equipment Usage and Refurbishment Schedule



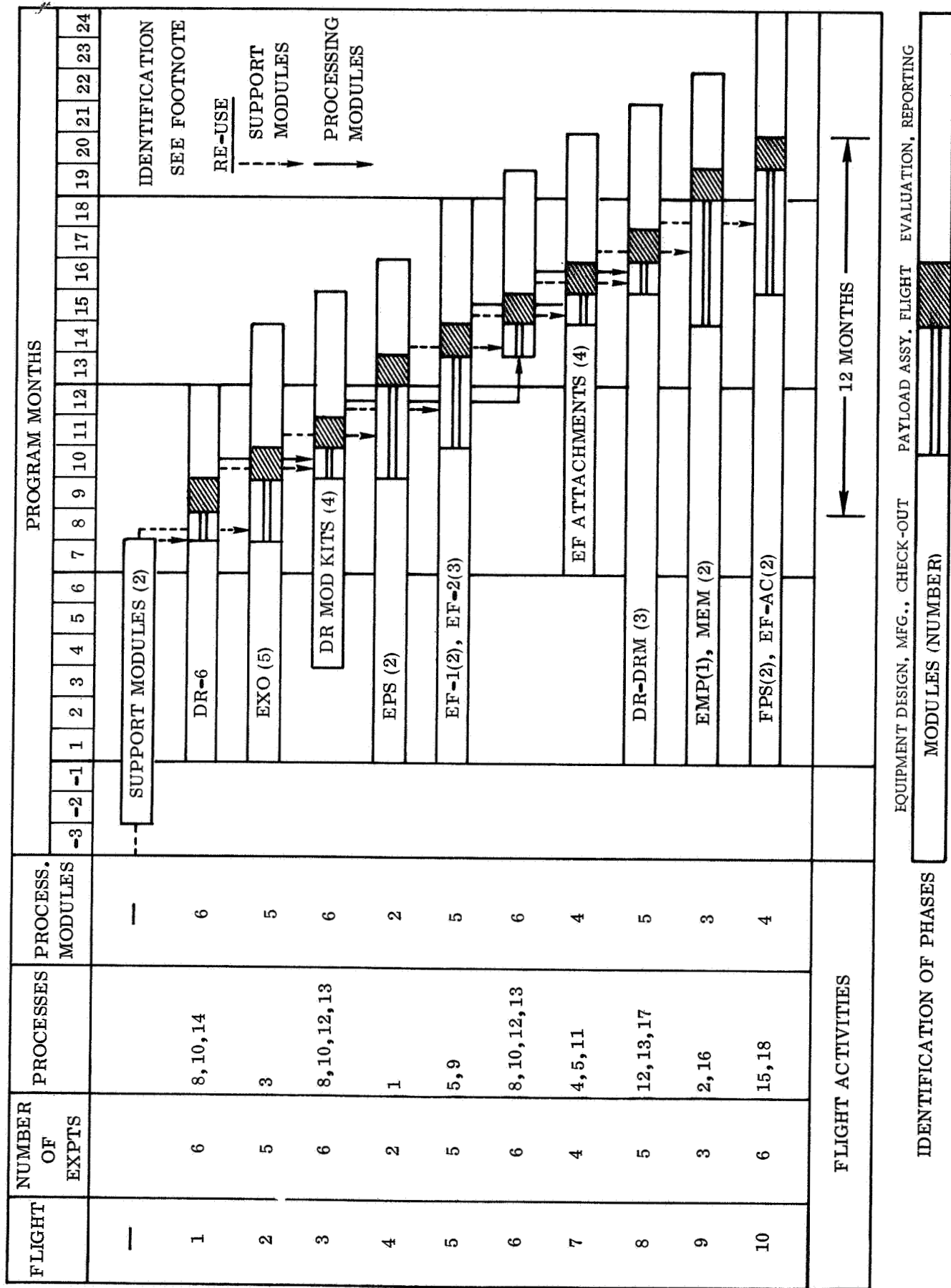
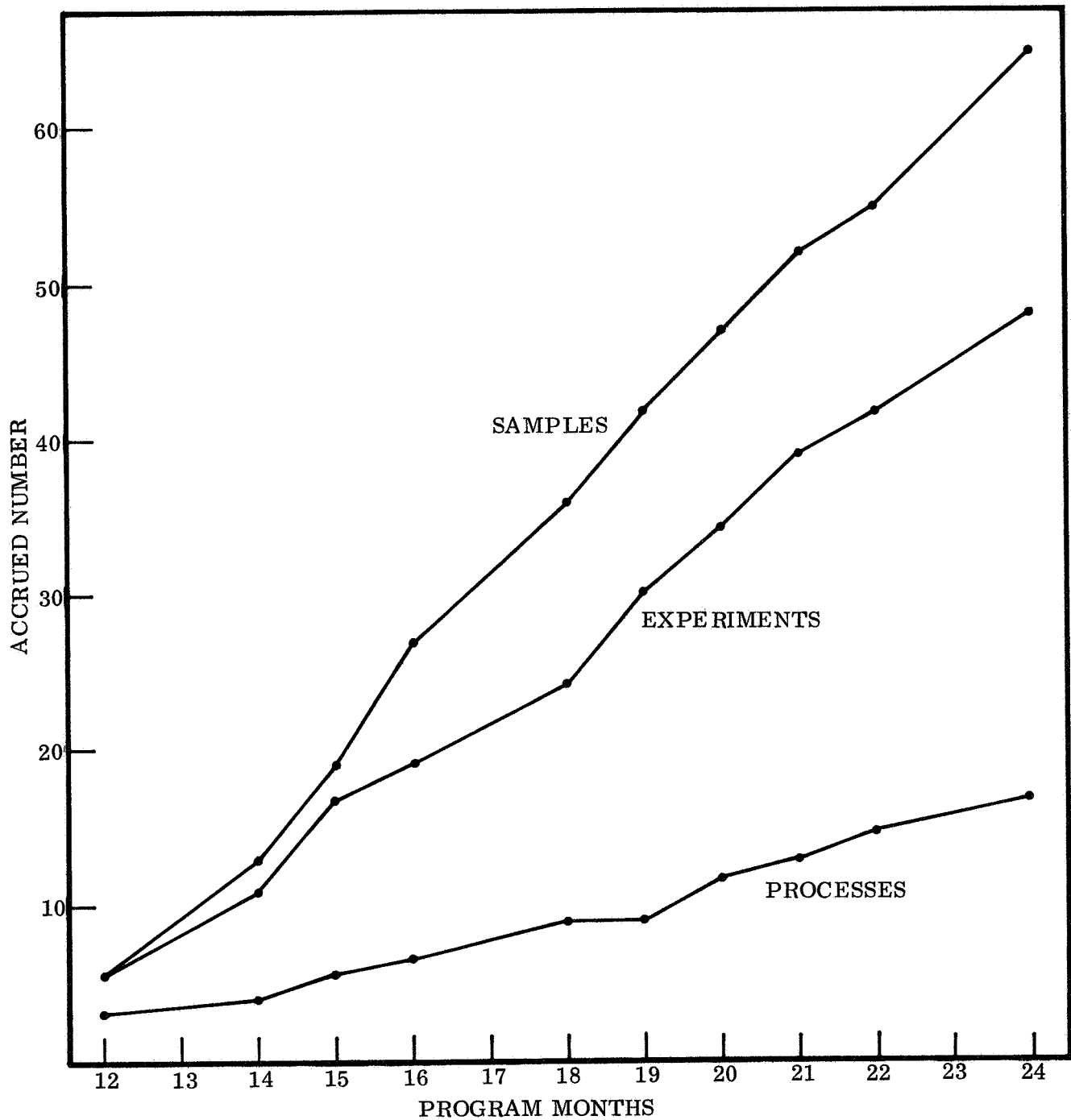


Figure 9-6. 10-Flight Program Schedule



Cumulative number of evaluated processes, experiments and samples vs. program months. (Includes time for evaluation and informal documentation).

Figure 9-7. Availability of Program Results

## 10. CONCLUSIONS

On the basis of the results of this study the following conclusions can be drawn:

1. Practically all basic space processes can be effectively and reliably verified in suborbital experiments.
2. The low-g time required for a complete process cycle and for material quantities which permit a conclusive evaluation ranges from 40 to 390 seconds. The required g-levels are in the order of  $10^{-3}$  to  $10^{-5}$  g. These requirements can be perfectly met with land-based trajectories of research rockets (sounding rockets). The use of such rockets and the related range operations are fully established within NASA.
3. Drop tower and aircraft low-g experiments are confined to the verification of specific process parameters and, for a limited number of processes, to an exploratory process evaluation.
4. The effectiveness of rocket experiments can be increased significantly by the use of multiple-experiment payloads. The number of experiments which can be accommodated on one research rocket flight ranges from 2 to 6, with an average of 4-5 experiments per flight.
5. The effectiveness and flexibility of a rocket test program is greatly enhanced by a modular equipment design, providing a high degree of equipment compatibility and interchangeability.
6. An initial verification of practically all typical space processes can be accomplished in a 10-flight test program requiring the acquisition of 5 research rockets. A detailed plan for such a program, assumed to represent the first phase of a continuing test program, has been established in this study. It comprises 17 processes and 48 experiments

(processing conditions), producing a total of 65 samples (material compositions) for evaluation.

7. The defined test program can be started at an early date in view of the advanced state of NASA-sponsored process developments which reduces the equipment lead times substantially.
8. The results of a rocket test program are expected to significantly increase the degree of confidence in the definition of experiments and facilities for shuttle-based space laboratories.